

# **Studies on Blast Furnace Slag Flow Characteristics**

*Thesis submitted to  
National Institute of Technology, Rourkela  
For the award of the degree*

*of*

**Master of Technology  
In  
Metallurgical and Materials Engineering**

*by*

**Vikas Saheb  
Roll No.: 210MM1156**



**DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING  
NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA**

**May-2012**

# **Studies on Blast Furnace Slag Flow Characteristics**

*Thesis submitted to  
National Institute of Technology, Rourkela  
For the award of the degree*

*of*

**Master of Technology  
In  
Metallurgical and Materials Engineering**

*by*

**Vikas Saheb  
Roll No.: 210MM1156**

*Under the supervision of*  
**Prof. U.K.Mohanty  
&  
Prof. S.Sarkar**



**DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING  
NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA**

**May-2012**



DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING  
NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA  
ORISSA, INDIA - 769008

---

## CERTIFICATE

---

This is to certify that the thesis titled *“Studies on Blast Furnace Slag Flow Characteristics”*, submitted to the National Institute of Technology, Rourkela by **Mr. Vikas Saheb**, Roll No. **210MM1156** for the award of Master of Technology in Metallurgical and Materials Engineering, is a bona fide record of research work carried out by him under my supervision and guidance.

The candidate has fulfilled all the prescribed requirements.

The Thesis which is based on candidate's own work, has not submitted elsewhere for a degree/diploma.

In my opinion, the thesis is of standard required for the award of a Master of Technology degree in Metallurgical and Materials Engineering.

**Prof. U.K. Mohanty**

Department of Metallurgical and Materials  
Engineering  
National Institute of Technology  
Rourkela – 769008

**Prof. Smarajit Sarkar**

Department of Metallurgical and Materials  
Engineering  
National Institute of Technology  
Rourkela – 769008

## ACKNOWLEDGEMENT

It is an honor for us to present this project which has helped us in enhancing our practical and theoretical skills in various metallurgical aspects. We wish to express our deep sense of gratitude to **Prof. B.C.Ray**, HOD, Metallurgical and Materials Engineering., NIT Rourkela for giving us an opportunity to work on this project.

I am highly indebted to **Prof. U.K.Mohanty**, **Prof. S.Sarkar** my guide, for his consistent encouragement, guidance and support to carry out and complete this project. I am also thankful to **Prof. S.K.Sahoo** for his sincere guidance and support without which the project would not have been successful.

I would be highly obliged to extend our thanks to **Mr. Uday Kumar Sahu** for his immense support and help rendered while carrying out our experiments, without which the completion of this project would have been at stake.

I would like to express my sincere gratitude to all the **faculty members and staff** of the department for their unflinching support, inspiration, and cooperation and providing me with all sort of official facilities in various ways for the completion of the thesis work.

I would also like to thank all my friends & my seniors, for extending their technical and personal support and making my stay pleasant and enjoyable.

At last but not the least; I remain really indebted to my family, **my parents** instilled strength especially at times when life was tough and supported me throughout my difficulty period with endurance.

With much love, I would like to thank **my younger brothers, sister and my bhaiya** and who with their kind and encouraging words provided me with strong moral support.

**VIKAS SAHEB**

# CONTENTS

<b>Abstract</b>	<b>I</b>
<b>List of Figures</b>	<b>II</b>
<b>List of Tables</b>	<b>III</b>
<b><u>Chapter-1</u> Introduction</b>	<b>1-3</b>
1.1. Introduction	1
<b><u>Chapter-2</u> Literature Survey</b>	<b>4-24</b>
2.1. Introduction to Blast Furnace Slag	4
2.2. Blast Furnace Operations	5
2.3. Blast Furnace Reactions	8
2.3.1. Reactions in the Upper Zone	9
2.3.2. Reactions in the Middle Zone	10
2.3.3. Reactions in the Lower Zone	10
2.4. Different zones of Blast Furnace	11
2.4.1. Cohesive Zone	11
2.5. Blast Furnace Slag	
2.5.1. General Overview	14
2.5.2. Forms of Slag	15
2.5.3. Slag Composition	16
2.5.4. Slag Structure	17
2.5.5. Slag Viscosity	18
2.5.6. Slag Liquidus Temperature	19
2.6. Effect of slag basicity on viscosity of the slag	20
2.7. Effect of MgO and Al <sub>2</sub> O <sub>3</sub> content on viscosity of the slag	22
2.8. Effect of slag basicity on the liquidus temperature of the slag	23
2.9. Cohesive zone model of blast furnace	23
2.10. Effect of silicon and its oxides on properties of slag and metal	24
<b><u>Chapter-3</u> Experimental Details: Theory and Procedure</b>	<b>25 - 51</b>
3.1. Flow Characteristics of Blast Furnace Slag	25
3.1.1. Initial Deformation Temperature (IDT)	25
3.1.2. Softening Temperature (ST)	25

3.1.3. Hemispherical Temperature (HT)	25
3.1.4. Flow Temperature (FT)	25
3.2. Experimental Apparatus	26
3.2.1. High Temperature Microscope	26
3.2.2. Planetary Ball Mill	27
3.2.3. Abrasion Tester Mixer	28
3.2.4. Sintering Furnace	28
3.2.5. Pelletizer Machine	29
3.2.6. X-ray Diffraction Machine	30
3.2.7. Scanning electron microscopic Machine	31
3.3. Experimental Work	31
3.4. Experimental Procedure	32
3.5. Experimental Result and Discussion	38
<b><u>Chapter-4</u> Conclusion</b>	<b>52 - 54</b>
Conclusion	52
Future work	52
References	53

## **Abstract**

The project aim set measurement of flow characteristics of blast furnace slag. The industrial slag (actual slag from different blast furnace), synthetic slag prepared in the laboratory for pure oxides as obtained from market and iron bearing materials with various extents of reduction resembling that expected to be in the cohesive zone as per literature are chosen for measurement while the final slag obtained from the industry reveals shortness of the slag. At C/S ratio of 1.04 to 1.09 and 10.4 to 11.01 MgO % the synthetic slag doesn't shown a clear trained, the iron bearing materials reveals variation of the characteristics temperature with variation in the extend of the reduction, SEM micrograph and XRD plots of the iron bearing material with different extended of reduction reveal compositional changes associated with structural changes.

Keywords: Blast furnace, Cohesive zone, Quality of hot metal, C/S ratio

# List of Figures

---

**Figure 1:** A schematic diagram of Blast Furnace Process

**Figure 2:** Schematic sectional diagram of the internal zones in a Blast Furnace.

**Figure 3:** Schematic diagram of Blast Furnace Operation and Blast Furnace Slag Production

**Figure 4:** Schematic diagram of the Silicate Tetrahedron in the Crystalline and Molten silica.

**Figure 5:** Pictorial view of Leitz Heating Microscope

**Figure 6:** Schematic diagram of the Heating Microscope

**Figure 7:** A four station Planetary Ball Mill

**Figure 8:** Abrasion Testing Mixer

**Figure 9:** Sintering Furnace

**Figure 10:** Pelletizer Machine

**Figure 11:** X-ray Diffraction Machine

**Figure 12:** Coning and Quartering

**Figure 13:** View of Different Flow characteristics of the slag prepared in the lab observed through Leitz Heating Microscope

**Figure 14:** Synthetic Slag prepared in the laboratory

**Figure 15:** Variation of Different Characteristic Temperatures with C/S ratio

**Figure 16:** Variation of (FT – ST) with C/S ratio

**Figure 17:** Variation of Different Characteristic Temperatures with MgO content

**Figure 18:** Variation of Characteristic Temperatures with C/S ratio for Slag nos. 1-5

**Figure 19:** Variation of ST-FT Temperatures with C/S ratio for Slag nos. 1-5

**Figure 20:** Variation of Characteristic Temperatures with MgO content for Slag nos. 6-10

**Figure 21:** Variation of ST-FT Temperatures with MgO content for Slag nos. 6-10

**Figure 22:** Variation of Characteristic Temperatures with MgO content for Slag nos. 11-15

**Figure 23:** Variation of ST-FT Temperatures with MgO content for Slag nos. 11-15

**Figure 24:** %Reduction Vs Characteristics Temperature

**Figure 25:** SEM Micrographs

**Figure 26:** The XRD plots for the 10% Reduction, 50% Reduction and 65% Reduction Samples



# List of Tables

---

**Table 1:** Chemical Composition of Industrial Blast Furnace Slags

**Table 2:** Characteristics Temperature of Industrial Blast Furnace Slag

**Table 3:** Variation of C/S ratio at fixed MgO percentage

**Table 4:** Variation of MgO percentage at fixed C/S ratio

**Table.5:** Variation of MgO (theoretical) at fixed C/S ratio

**Table 6:** Characteristics Temperature of Slags presented in Table ‘3’

**Table 7:** Characteristics Temperature of Slags presented in Table ‘4’

**Table 8:** Characteristics Temperature of Slags presented in Table ‘5’

**Table 9:** Chemical analysis of Iron ore and other raw materials

**Table 10:** Chemical composition of pellets after curing at 950°C for two Hours

**Table 11:** %Reduction Vs Characteristics Temperature

# CHAPTER-1

---

## INTRODUCTION

## CHAPTER-1 INTRODUCTION

### 1.1. Introduction

Blast furnace route of Iron making will probably remain the major process for pig-iron production, to be finally converted to steel, despite the speedy deflection of coking coal reserves in India and elsewhere in the world. It is thus important to understand and study the factors responsible for influencing the smoothness of operation, coke consumption, and quality of hot metal produced and over all, the associated green-house problem.

The Blast furnace process runs on the principle of counter current flow, the down coming solid charge meeting the up going hot gasses consisting mostly of reducing gasses and nitrogen. Obviously the gas-pressure, temperature and composition of the burden greatly influence the process. While most of the charge material soften and melt at relatively higher up the furnace the coke lumps stay solid and are responsible for keeping the bed open even at the tuyer zone. On the other hand the softened/melted solids affect the permeability of the bed and restrict the gas flow, allowing the flow of gas only through coke-slits. This result in a pressure drops and influences the gas-solid-liquid interface greatly influencing the slag metal reaction rates and thus the blast furnace process of iron making.

The blast furnace can be roughly divided into a number of zones out of which the granular zone, the cohesive zone, the bosh and the hearth are important. The cohesive zone from the point of view of gas flow and permeability of the bed is of paramount importance. This zone is below the granular zone where the up going gasses containing CO initiate the reduction of the iron oxide in the ore. This reduction is termed as indirect reduction in the sense that the reduction is carried out by 'CO' a product of 'C' and 'O<sub>2</sub>' and not by 'C' directly. This is an exothermic reduction unlike the direct reduction which is endothermic. Obviously a higher proportion of indirect reduction in the blast furnace would decrease the coke consumption as a fuel, responsible for heat generation.

The cohesive zone in the blast furnace is bound by softening of the iron bearing materials at the top and flow/liquid mobility of the same of its bottom end. Therefore that the granular zone is extended and that the proportion of indirect reduction is enhanced, the cohesive zone should form lower down the furnace. This means the softening temperature of the iron leaving materials should be higher pushing the cohesive zone down the furnace. Obviously

the prevailing composition, pressure and temperature conditions should result in high softening temperature. Once this is achieved, it is important to note that the cohesive zone should be narrowed down. This means the lower end of the zone should move towards the upper end. i.e., the flow temperature of the charge should be relatively lower. Again the composition of the charge as affected by the temperature and pressure conditions prevailing is the key factor. This lowering of the narrow cohesive zone would lessen the distance of traverse of hot metal before it reaches the bosh and would have a retarding effect in the reaction between the ascending gases (containing SiO) and the descending metal droplets, thus retarding the silicon peak by the hot metal improving the quality of hot metal vis-a-vis the subsequent steel making process.

The blast furnace slag which comes out of the furnace is the final slag and would not reflect the prevailing conditions in the cohesive zone. The cohesive zone slag on the other hand, can not be obtained. Therefore, in order to analyse the cohesive zone slag pertinent that the slag resembling that in the cohesive zone, as presented in literature, may be synthetically prepared in the laboratory.

Literature study reveals that the iron bearing material enters the cohesive zone with reductions in excess of 60 percent. The first liquid formed as a consequence of reaction between the gangue and the reduced iron ore (wustite), is enveloped by a porous iron shell. This shell, on descents, meets severe conditions of pressure and temperature and also gets carburised. Its melting point is lowered and under pressure it crumbles allowing the liquid at its core to be exposed to the outside.

This can be simulated in the laboratory. Pellets can be prepared and subjected to different extents of reduction and these can be examined to measure the temperature at which it soften/melts. The effect of composition on the softening/melting temperature can be recorded. Such a study would throw light on the relationship between the characteristic temperatures concerning softening/melting and the composition which in its turn can be used to advantage to influence the process of iron making, the coke consumption and the quality of the hot metal produced.

Keeping the above in mind, the present work performs the experiments in three phases. The first phase involves the final slag from the blast furnace. The characteristic temperatures revealing the composition at which a short-slag is produced. This slag flows as soon as it softens, leaving the site and trickling down to extend the slag metal interface. The second phase of the experiment involves synthetic slag prepared in the laboratory from pure oxides. These slags resemble the best composition as obtained in the first phase of operation. The third phase involves iron ore pellets prepared from raw ore, flux and coke with a suitable binder (2% bentonite in this case), subjecting it to different extents of reduction to in line with that happening in the cohesive zone as per literature.

An attempt is made to perform an exhaustive study of these three, namely the final slag, the synthetic slag and the reduced iron ore pellet vis-a-vis the characteristic softening and melting temperatures and correlating same with the same chemical composition.

# CHAPTER-2

---

## LITERATURE SURVEY

## CHAPTER-2 LITERATURE SURVE

### 2.1. Introduction to Blast Furnace Slag

Slag making has always been a vital part of steel and iron making. After all, at least 10% of the slag was produced in the steel and iron making process. In ancient times, the secret of steel and iron making is in the slag formulations. Today's iron maker has a better understanding the importance of blast furnace slag compositions and their affects on hot metal economics and quality. There is an old saying in iron making "Take care of the slag and it will take care of the metal". For making a good quality of hot metal, slags are a very important part in iron making. They remove unwanted oxides, sulphides, nitrides and phosphides. However slag compositions must be closely controlled or else they will cause additional problems and become liabilities and the improper slag compositions can lead to harmful reversion of S, P, Si and various oxide inclusions.

In order to make blast furnace slag practicable and more effective, completely homogenous liquid at the operating temperature must be taken. It must also have sufficient fluidity so that it can easily run out of the hearth without any loss of production time. In addition, following are the properties that the slag must ensure (For getting a good quality of hot metal) [2], [3]

- Removal of impurities
- Best possible slag-metal reaction
- Less metal entrapment through better slag-metal separation.

And for smooth operation of any blast furnace, the slag must satisfy the following requirements: [1]

- The slag volume should be kept as low as possible.
- It should have the properties of alkali removal and fulfil the desulphurisation requirements.
- The composition of the primary slag must be uniform.
- Slag formation should be confined to a limited height of blast furnace and the slag should be stable.
- The slag should provide good permeability in the zone of slag formation.
- The melting point of the slag should be neither too high nor too low.

In blast furnace, as the burden descends down inside the furnace, the quality of hot metal obtained is dependent on the formation of the slag and its mineralogical transformations. It is well known that the components of slag namely silica and alumina increase the viscosity whereas the presence of calcium oxide reduces the viscosity. The melting zone of slag determines the cohesive zone of blast furnace and hence the fluidity and melting characteristics of slag play a major role in determining the blast furnace productivity. Initially iron rich slag is formed and thereafter due to assimilation of CaO and MgO from flux, the composition of slag varies. As the slag trickles down, it assimilates silica and alumina of ash, generated from combustion of coke. The process of trickling down depends on viscosity of slag, which further is governed by the composition and temperature of the melt.

## **2.2. Blast furnace operations**

In the blast furnace plant iron oxide comes in the form of raw ore, pellets or sinter. The raw ore is removed from the earth and sized into pieces that range between 0.5 to 1.5 inches. The ore consist of almost 50-70% of iron therefore; the ore may be either Hematite ( $\text{Fe}_2\text{O}_3$ ) or Magnetite ( $\text{Fe}_3\text{O}_4$ ) and can be charged directly into a blast furnace without any further processing. It is beneficiated to increase the iron content of low iron containing ore by purification process. Waste material can be removed from the ore by crushing and grinding it into a powder called gangue. Pellets can be created from this low iron content ore formed by the remaining iron-rich powder which is rolled into balls and fired in a furnace. This process lead to the production of strong, marble-sized pellets containing of about 60% to 65% iron.

Fine raw ore, small coke, sand-sized limestone and numerous other steel plant waste materials containing some iron are required for the production of sinter. These materials are fine and mixed together in equal proportion and then placed on a sintering strand, which is similar to a steel conveyor belt. In sintering strand mixture is ignited by gas fired furnace and fused by the heat from the coke fines into larger size pieces ranges from about 0.5 to 2.0 inches. The liquid iron produced in the blast furnace from ore, pellet and sinter with some of the remaining impurities going to the liquid slag.

The coke consist 90 to 93% carbon, some ash and Sulphur produced from the mixture of the coal. Coke is produced by crushing and grinding of coal into powder and then charged into an oven. After 18 to 24 hour of reaction time most of the volatile matter get removed and the cooked coal which is called as coke; present in the furnace. Before that screening of the cooled coke is done, their ranging from 1 to 4 inches. Since the strength of the coke is more



than that of raw coal. The high energy value of strong pieces of coke is responsible for good permeability, heat and gases which are mostly required for reduction and melting of the iron ore, pellets and sinter.

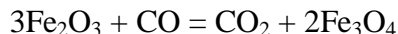
The raw material “limestone” extracted from the earth by blasting with explosive and used in the iron making process. It is then fragmentize and analyze to a size ranging from 0.5 to 1.5 inch to form flux for the blast furnace operation. These fluxes are blend of two types of lime stone containing pure high calcium limestone and dolomite limestone.

Since the limestone is melted to form slag which removes Sulphur and other impurities. The blast furnace operator may blend the different stones to bring out the desired slag chemistry and produce optimum slag carrying low melting point and a high fluidity etc.

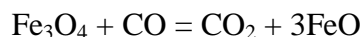
Before charging all the raw materials stored in an ore field then transferred to the stock house. Once these materials get charged, the numerous of chemical and physical reactions occur in the furnace while descending towards the bottom of the furnace.

The reduction of iron ore, pellet and sinter takes place by a series of the chemical reactions. These reactions are:

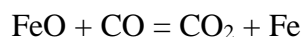
➤ At 850° F



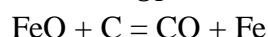
➤ At 1100° F



➤ At 1300° F



Or

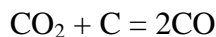


Softening and melting of iron occurs after the reduction reaction of iron ore, which finally trickles down as the liquid iron through the layers of coke present at the bottom of the blast furnace.

Then the coke goes down to the bottom of the furnace, to the level where the preheated airs get in the furnace. From that hot blast, coke is ignited and it immediately reacts to the air and induces heat, as shown in the reaction below:



Due to the presence of excess carbon, the carbon dioxide was reduced to carbon monoxide at a high temperature. There reaction as follows:

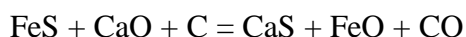


The product obtained from the above reaction; carbon monoxide is required for indirect reduction of iron oxide as seen in the preceding iron oxide reactions.

Limestone and dolomite, if added as a flux in the charge, as the charge descends down in the furnace it stay in a solid state while going through its first reaction takes place, as shown:



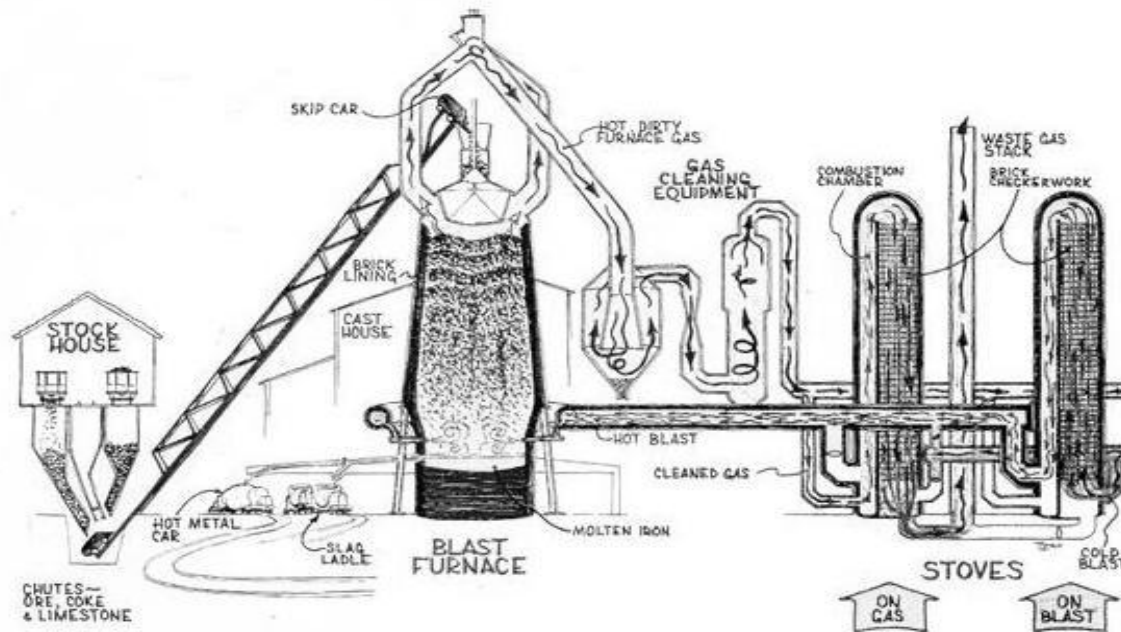
This reaction necessitates energy and starts in the temperature zone of 800-1000<sup>0</sup>C. The resulting basic oxides combine with gangue oxides to form slag and the CaO formed from this reaction is used to withdraw sulphur from the iron which is essential before the hot metal becomes steel. This sulphur removing reaction is:



These CaS becomes part of the slag which can be also formed from any remaining Silica (SiO<sub>2</sub>), Alumina (Al<sub>2</sub>O<sub>3</sub>), Magnesia (MgO) or Calcia (CaO), alkali-alkaline earth oxides, etc. with some minor other oxides and sulphides that entered with the iron ore, pellets, sinter or coke. The liquid slag due its low density trickles down through the coke bed to the bottom of the furnace where it floats over the molten iron.

In addition to molten iron and slag, another product of the iron making process is hot dirty gases which comes from the top of the blast furnace and passed through gas cleaning equipment where particulate matter was removed from the gas and then gas is cooled. These gasses are burned as a fuel in the “hot blast stoves” because of the presence of considerable energy value and are also used for preheating the air entering the furnace known as hot blast. The unburnt gases are sent to the boiler to generate steam which rotates a turbo blower and

generates the compressed air called as a "cold blast" that comes from the stoves. [1], [2], [3]



*Fig. 1-A schematic diagram of Blast Furnace Process*

In brief we can say that the blast furnace operates as a counter current process in which the downward flow of the solids occur with upward flow of the hot gases. Numerous of chemical and physical reactions take place throughout the furnace to produce desired final product known as hot metal. The typical hot metal chemistry follows <sup>[4]</sup>:

Iron (Fe)	=	93.5-95.0%
Silicon (Si)	=	0.30-0.90%
Sulphur (S)	=	0.025-0.050%
Manganese (Mn)	=	0.55-0.75%
Phosphorous (P)	=	0.03-0.09%
Titanium (Ti)	=	0.02-0.06%
Carbon (C)	=	4.1-4.4%

### 2.3. Blast Furnace Reactions

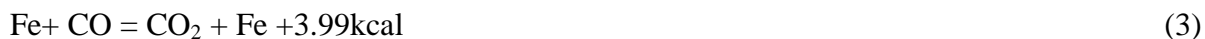
Iron blast furnace is used for reduction and smelting of iron ore to produce industrial metals. In this furnace; fuel (coke), ore, flux are continuously supplied to the top of the furnace. Preheated air are blown from the bottom of the furnace into the coke bed and generate heat & reducing gas carbon monoxide, required for smelting of the iron ore and to produce liquid

iron. The slag and the metal form two separate layers at the bottom of the blast furnace. In order to change, the reactant into product various stoichiometry must be met, moreover endothermic reactions proceed to maintain the product to their final temperatures. The blast furnace can be conveniently divided into three zones for a study of the physical and chemical reactions occurring in them. The zones are shown below:

- The upper or pre-heating or preparation zone.
- The middle or indirect reduction or thermal reserve or isothermal zone.
- The lower or processing or melting or direct reduction zone.

### 2.3.1. Reactions in the Upper Zone

Several chemical reactions are taking place in the blast furnace out of which the reduction reactions of iron oxide are mainly concern. The product form after the reduction of iron ore is well known. The greater the driving forces for reduction and the faster the rate of chemical reaction, the product layer formation is more pronounced.



Simultaneously iron oxide going through this purifying reaction, begin to soften, melt and finally trickle as a liquid iron to the bottom of furnace through the coke. At temperature of about 700-900°C, magnetite is reduced to wustite and thermodynamically deposition of carbon occur by carbon monoxide reaction.



The hot blast temperature can be from in temperature range of 440-600°C where the deposition occur, in the presence of iron and its oxides act as catalyst in the reaction. Carbon monoxide is act as reducing agent and reduces H<sub>2</sub>O in the upper zone of the furnace up to a certain level.



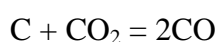
At a low temperature of about 400°C decomposition of carbonates (other than those of calcium) occurs. Nowadays modern furnace is not very important because they are using pellets and sinter and these carbonates are calcined from outsides.

### 2.3.2. Reactions in the Middle Zone

It is a moderate temperature zone where the temperature ranges between 800 to 1000° C. Most of the indirect reduction of wustite is (equation 3) take place in this zone. The ratio of CO/CO<sub>2</sub> gas is 2.3, a value exhibiting equilibrium with Fe-FeO (Eq. 3). The indirect reduction will be more if the height of this Zone (800-1000° C temperature zone) is large since the contact time is longer between gas/solid. In brief the rate of reduction of ore in the middle zone must not be in a restriction for the attainment of optimum coke rate.

### 2.3.3. Reactions in the Lower Zone

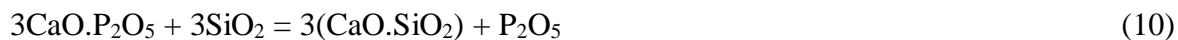
A variety of physical and chemical processes occurs in the lower zone consisting the temperature of about 900-1000° C. Unreduced iron oxide moves into this zone as calcium ferrites or fayalite, in the primary slag. At above 1000°C direct reduction reaction of iron oxide takes place according to:



A very high temperature is required for the reduction of Si and Ti, while the oxides of Mg, Ca and Al are highly stable such that they are reduced to a negligible amount. At the high temperature the reduction of Mn from its monoxide takes place which is quite difficult and Cr and V behaves in similar manner as Mn.



Phosphorus is present in the form of tri- or tetra- phosphate of Calcium. Silica helps in the cleavage of the phosphate bond.



## 2.4. DIFFERENT ZONES OF BLAST FURNACE

The significance of melting and softening features of the blast furnace was established in late 17s in Japan by the data collected from the dissection of numerous quenched blast furnace operations. The dissection studies revolutionized the understanding of blast furnace process by providing detailed accounts of physical and chemical phenomena taking place inside the furnace. It was found that internal state of furnace comprises of five distinctly different yet highly interrelated zones. The zones are given below:

- Granular zone/isothermal zone
- Cohesive zone
- Active coke zone
- Stagnant coke zone
- Hearth

Out of five zones the cohesive zone plays the most important role in the B.F. operations. Consequently by the above studies it was found that at the temperature range of about 1000 to 1500°C the existence of cohesive zone was achieved.

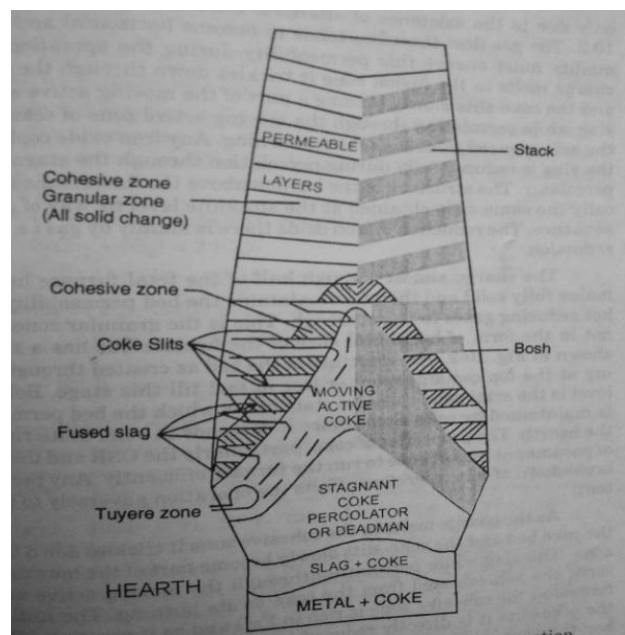
The shape and location of this zone affect the blast furnace productivity, fuel consumption, hot metal quality, operational stability and lining life. The pattern of cohesive zone was found out to be a function of iron bearing material properties. This provides a great significance for analyzing softening and melting phenomena and therefore characteristics of the iron bearing material were considered to be important.

### 2.4.1. Cohesive zone

As the burden descends further, and its temperature rises on account of contact with the ascending hot gases, softening and melting of the iron-bearing solids takes place in the so-called cohesive zone (mushy zone). The cohesive zone in a blast furnace, bound by the softening line at the top end and the melting line at its bottom end acts as a gas distributor in

the blast furnace. The cohesive zone determines gas flow pattern. The location of the cohesive zone's (the height above the tuyere level) as well as its thickness depends on the softening and melting characteristics of the burden of the blast furnace. The blast furnace being basically a counter current process, the distribution of gas as influenced by the gas-semi liquid/ gas-liquid permeability greatly affects the BF operation particularly influencing the productivity, hot metal quality and coke consumption both as a fuel and a reducing agent. The ore is compacted due to softening and melting in the cohesive zone. Minimizing the size, lowering the level of the cohesive zone, increases the efficiency of the blast furnace, improves productivity and decreases coke rate. The thickness and position of cohesive zone affects the following things:

- Gas permeability
- Extent of indirect reduction
- Si content of the pig iron



**Fig2:** Schematic sectional diagram of the internal zones in a blast furnace.

#### 2.4.1.1. Gas permeability

Gas permeability deals with thickness of cohesive zones, and the thickness of the cohesive zone depends on the softening and melting characteristics of the burden of the blast furnace. Cohesive zone consist of alternative impermeable, gas-semi liquid/ gas-liquid layers resisting

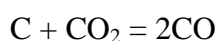
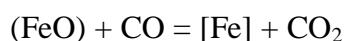
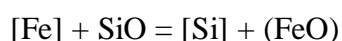
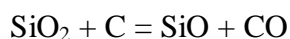
the flow of ascending gases. . If the more and more gases are allowed to pass through coke slits and the active coke zone (dripping zone) then thickness of cohesive zone is less. The wind volume depend on the length of the coke slit, viscosity and band volume of the melt and their resulting influence is loss of permeability of the bed and the cohesive zone has the lowest permeability.[12],[13]

#### **2.4.1.2. Extent of indirect reduction**

Extend of indirect reduction depends on the location of the cohesive zone. For proper gas flow and an increased extend of indirect reduction it is essential that the cohesive zone form lower down the blast furnace in other words it means that the softening temperature of the blast furnace slag should be high. If the position of cohesive zone is deeper into the blast furnace, since the volume of the granular zone is increased, and the descending materials stay more time in the granular zone. Thus the carbon monoxide is better utilized to reduce the wustite indirectly. It means, increase the volume of granular zone improving the gas utilization in terms of indirect reduction. Since, the extent of indirect reduction is increased in a lower positioned cohesive zone and by lowering of the cohesive zone; the coke consumption for the production of one tone of pig iron gets highly reduced.

#### **2.4.1.3. Si content of the pig iron**

Pig iron constitute of Si which is form due to the reduction of SiO produced by reduction of SiO<sub>2</sub>. The reduction of SiO takes place in the dripping zone or bosh region and present in ash after burning of coke near the raceway. The resulting in a decrease in the dripping zone, thereby decreasing the contact time of metal droplets with SiO bearing gasses, resulting in low Si pick up in the melt.[25]





## 2.5. Blast Furnace slag

### 2.5.1. Overview

In blast furnace, as the burden (iron ore, iron scrap, fluxes (limestone and/or dolomite), and coke) descends down inside the furnace, the quality of hot metal obtained is dependent on the formation of the slag and its mineralogical transformations.

The Slag is a low melting chemical compound formed by the chemical reaction of the gangue and the flux in the charge. All unreduced ones (silicates, alumino-silicates, and calcium-alumina-silicates) join the slag. The major constituents of the slag include the following:

Major elements are

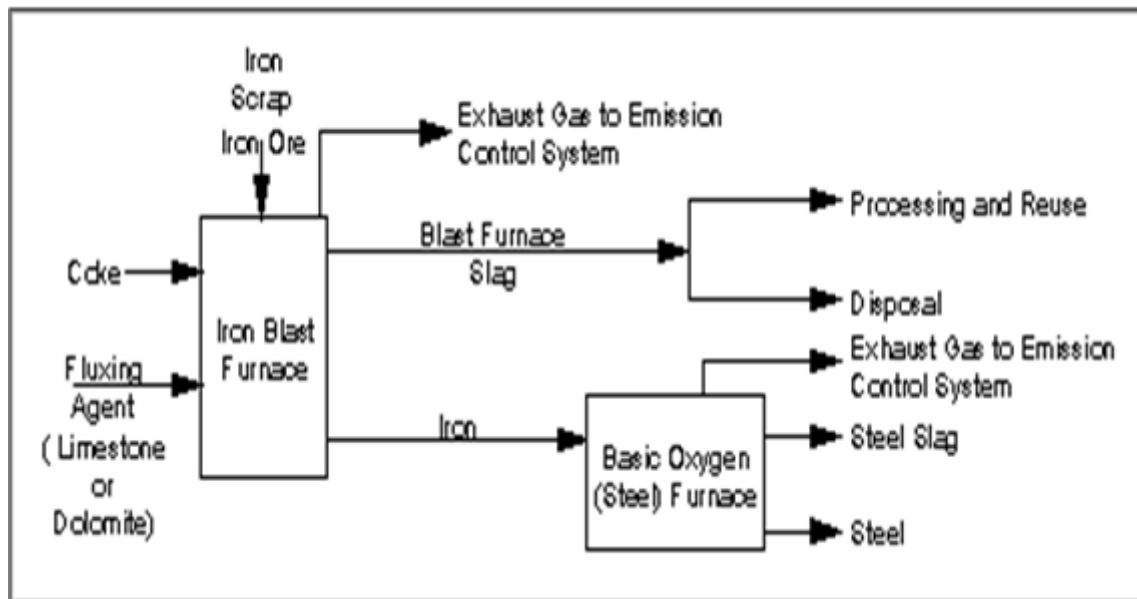
- $\text{Al}_2\text{O}_3$  – 20.45%
- $\text{CaO}$  – 32.23%
- $\text{SiO}_2$  – 33.02%
- $\text{MgO}$  – 9.95%

Minor elements are

- $\text{S}$  – 0.89%
- $\text{MnO}$  – 0.54%
- $\text{TiO}_2$  – 1.01%
- $\text{FeO}$  – 0.41%
- $\text{K}_2\text{O}+\text{Na}_2\text{O}$  – 1%
- Trace Oxides – 0.5%

It is said that the art of slag making is the art of iron making. The slag must have the affinity for absorbing impurities i.e. gangue from charge along with other deleterious impurities which affect the quality of hot metal. The blast furnace slag is essentially low melting compound consisting of silica, alumina, calcia and magnesia. The other minor constituents of slag depend on the quality of ore which includes manganese oxide, sulphur, phosphorous and compounds thereof. It is essential to know the behavior of slag in terms of the chemical composition, the mineralogical constitution and its ability to react and trap the minor impurities. Also the slag should be free flowing at the operating temperature with high slag metal separation with out entrapping metal. As the various properties of the final product are directly influenced by the composition of the slag. Thus the physicochemical properties of

slag play an important role in iron making operations. In this work, different physicochemical properties of different slag samples were measured and compared. Since the slag should have a low fusion temperature so that at the furnace operating temperature a high available super heat will increase the kinetics of chemical reaction will ensure better slag metal separation from both physical and chemical point of view. Fig. 3 shows a general layout of blast furnace operation which represents the blast furnace feed stocks and the production of blast furnace co products (iron and slag). [8]



*Fig. 3-Schematic diagram of blast furnace operation and blast furnace slag production*

### 2.5.2. Forms of slag

#### Air-Cooled Blast Furnace Slag

It is one of the various slag products, formed when the liquid slag is allowed to pour into beds and cooled under the atmospheric condition. A slag produced is hard, lump and crystalline in structure, which can subsequently be crushed and screened.

#### Expanded or Foamed Blast Furnace Slag

The process of cooling and solidification can be accelerated if the molten slag is cooled and solidified by adding controlled quantities of water, air, or steam. It enhances the cellular nature of the slag and produces a lightweight expanded or foamed product. Its relatively high porosity and low bulk density helps us to distinguish foamed slag from air cooled blast furnace.

**Pelletized Blast Furnace Slag**

Pelletized blast furnace slag has been used to granulate blast furnace slag in which less water is used with violent impelling of the viscous, but rapidly cooling slag through the air. The pellet can be made more crystalline by controlling the process and can be prove beneficial for aggregate use and vitrified. This vitrified pelletized blast furnace slag used for the production of cement.

**Granulated Blast Furnace Slag**

If the molten slag is cooled and solidified by rapid water quenching to a glassy state, crystallization can be avoided resulting in the formation of sand size (or frit-like) fragments, usually with some friable clinker-like material. According to chemical composition of the slag, its temperature at the time of water quenching, and the method of production are the three factors responsible for the physical structure and gradation of granulated slag. Ground granulated blast furnace slag (GGBFS) shows cementitious properties, when it is crushed or milled to very fine cementsized particles.

**2.5.3. Slag composition**

20 % of the total mass in iron production is formed into slag. Different Slags are a result of different cooling methods applied. Basicity ratio in a range of 1.25 to 1.55 is a result of calcium and magnesium content. The slag chemistry of overall components reported as equivalent to Calcium oxide plus magnesium oxide divided by silica is given below:

Major elements are

- CaO - 38%
- SiO<sub>2</sub> - 36%
- MgO - 10%

Minor elements are as follows:

- Iron Oxide (FeO & Fe<sub>2</sub>O<sub>3</sub>) - <1%
- Sulfur(S) - 1%
- Alumina (Al<sub>2</sub>O<sub>3</sub>) - 6-12%.

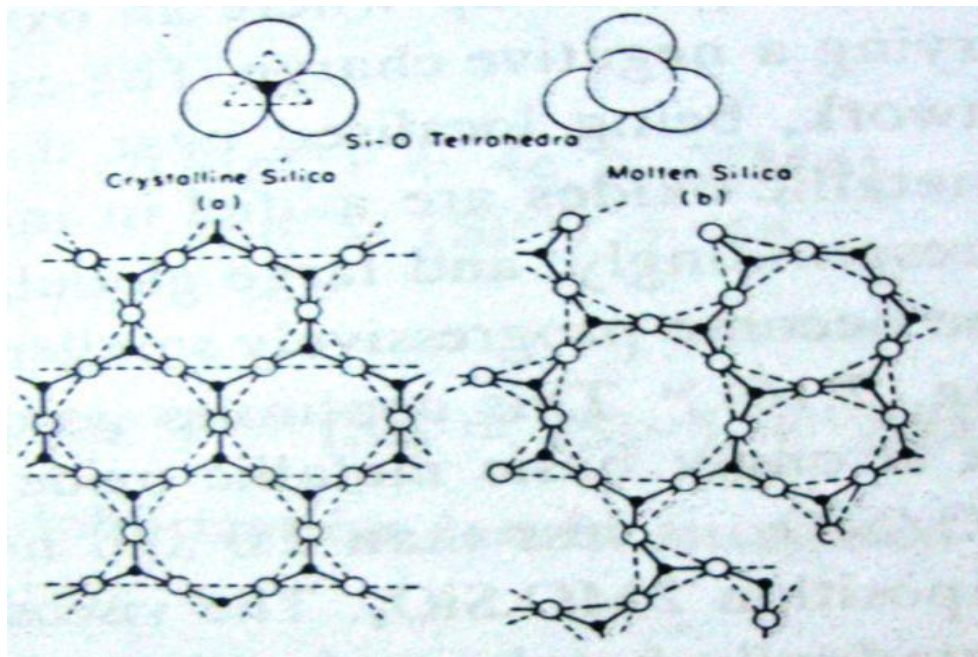
#### 2.5.4. Blast furnace slag structure

Liquid slags are homogenous melts consisting of oxides of silicon and other elements. They are known to possess electrical properties and consist of simple and complex ions. Crystal analysis of solid silica shows that silicon occupies the centre of a tetrahedron surrounded by 4 oxygen atoms, one at each of the four corners. Each oxygen atom is bonded to two silicon atoms and the network is continuous in three dimensions. These tetrahedra can share only corners so that when every corner oxygen atom is shared, the substance formed will have an overall stoichiometric formula of  $\text{SiO}_2$ . A Si atom has 4 valences. As each oxygen atom of the tetrahedron has a residual valence, therefore, the  $\text{SiO}_4$  group carries 4 negative charges, i.e.  $(\text{SiO}_4)^{4-}$ .

In the crystalline state the tetrahedral arrangement of the silicon and oxygen atoms is symmetrical. The solid structure does not undergo any sudden change on fusion, as expected. In molten or vitreous silica the structure becomes distorted but most of the corners remain shared. The viscosity of molten silica is very high ( $\approx 10^5$  P), the corners being linked tightly in all directions in a vast network.

The group,  $(\text{SiO}_4)^{4-}$  which is regarded as individual tetrahedron with silicon at the centre and oxygen at the four corners, can be assumed to exist as ion in the complex silicates. Measurement of the energy of activation for electrical conductance and other results indicate that the addition of CaO, MgO or other metal oxides to molten silica results in the breakdown of the three dimensional silicon-oxygen network into silicate ions. The driving force for the breakdown process is the attraction between silicon and oxygen. This depends on their relative valences and ionic radii.

When lime or Magnesia is added to molten Silica, two silicon-oxygen bonds are opened up giving rise to an open shared corner where oxygen is added, each oxygen carrying a negative charge. The cations distribute in the interstices of the network, being localized near the charged oxygen. As the metallic oxides are added in increasing amounts, the Si-O bonds break correspondingly, and large globular or ring type silicate ions are formed. These become progressively smaller as the metal oxide content increases as their no goes on increasing. As the strong silicate networks break, the viscosity of the melt decreases drastically as viscosity of a material depends not only on its composition but also on its structure. The more relaxed the structure is the less is the viscosity. [2]



**Fig. 4-** Schematic diagram of the silicate tetrahedron in the crystalline and molten silica. The oxygen and silicon atoms are presented by white and black respectively.

#### 2.5.5. Slag Viscosity

The blast furnace slag behaves as a Newtonian fluid due to the presence of range of shear stresses applicable on the slag. Generally the viscosity of Newtonian fluid does not depend on the presence of shear stress. It's the ionic and molecular structure that governs the viscosity of the blast furnace slag. Various important phenomena such as the heat transfer, mass transfer and the chemical reactions depend on the flow phenomena of the slag hence on its viscosity [20]. A comprehensive slag-metal separation depends on the free flow nature of the slag at operating temperatures. The gangue particles should go into the slag to ensure a metal of desired quality. The diffusion of ions to and from the slag to the metal influences the reaction rates and that too is very much dependent on the viscosity of the slag. Yet, from the available heat (in the hearth) point of view the blast furnace slag should be neither very viscous nor very fluid. Slag viscosity is a transport property that relates to the reaction kinetics and the degree of reduction of the final slag. The viscosity of the slag controls the aerodynamics such as the gas permeability and the heat transfer this in turn affects the efficiency of the blast furnace.

With the increase in basicity the 3-dimensional silicate network chains are broken into discrete anionic groups hence, the viscosity of the slag decreases. Beyond a certain level of

basicity the chemical potential of certain solid phases increases and there's also an increase in the viscosity of the slag.

The slag-metal separation efficiency, the metal quality now can be explained and based on the viscous nature of the slag. The ease with which the slag can be tapped depends on its viscosity and hence the energy requirement and the profitability of the process. The slag viscosity is sensitive to its ionic and molecular structure.

#### **2.5.6. Slag Liquidus Temperature**

The liquidus temperature can be defined as the temperature at which the slag when heated it assumes a hemispherical shape according to the German Industrial Standards 51730. The slag sample observed having a hemispherical form is of a very small mass. This temperature can also be nominated as the temperature at which the first crystal is formed when the melt is cooled down as indicated by Osborn [21] and Snow [22]. While Ohno et al [23] indicated that all the crystal disappear when the slag is heated to the temperature of Liquidus temperature. So these are some of the numerous nomenclatures reported by scientists to indicate the liquidus temperature. In our experimental work the liquidus temperature is measured by the Leitz heating microscope.

The liquidus temperature gives us an approximated assumption of the position, width and shape of the cohesive zone. As the cohesive zone geometry affects the gas permeability along with the amount of silicon picked-up by the hot metal and also an idea about the extent of indirect reduction. Thus the liquidus temperature measurement bears a great importance for its measurement.

In a pyrometallurgical process or in industrial melts, the fusion behavior of the non-metallic melts i.e. the combination of oxides which form the slag plays an important part than the actual fusion temperature. The fusion behavior is described in terms of four characteristic temperatures. These are; the initial deformation temperature (IDT), symbolizing the surface stickiness; the softening temperature (ST), symbolizing the plastic distortion; the hemispherical temperature (HT), which is also the liquidus temperature, symbolizing sluggish Flow; and the flow temperature (FT), symbolizing liquid mobility.

The various physicochemical properties of slag play a vital role in iron making as this influence the final hot metal quality and the process economy. These properties not only influence the blast furnace operations but also increase the value of the slag as a by-product. The impurities entering the blast furnace in the form of their various compounds are removed in the slag phase. The presence of these compounds of the impurity items affects the slag structure and other properties. The slag must ensure efficient removal of impurities along with smooth running of the blast furnace processes. From this point of view, the slag characteristics are of prime importance in iron making operations. The most important physicochemical properties of slag, which have attracted the attention of many metallurgists, are: Slag Viscosity, Slag Liquidus Temperature, and Sulphide Capacity of the Slag and Alkali Capacity of the Slag.

## **2.6. Effect of slag basicity on viscosity of the slag**

**Y.S. Lee et al [4]** studied and observed the viscous behaviour of  $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3\text{-MgO-FeO}$  slag under controlled conditions of  $\text{C/S} = 1.15\text{-}1.6$ ,  $10\text{-}13\text{mass\% Al}_2\text{O}_3$ ,  $5\text{-}10\text{mass\% MgO}$  and  $0\text{-}20\%$   $\text{FeO}$ . The study of such a slag by the scientists leads them to infer that there's depolymerization of silicate network above the  $\text{C/S}$  ratio of 1.3 and less than the ratio of 1.5 this causes the viscosity of the slag to increase. The slag viscosity otherwise up till the ratio of 1.3 decreases due to the increasing chemical potential of the dicalcium silicate which is a primary solid phase. This good correlation between the viscosity and the slag components was basically due to the thermodynamic approach taken up for the activity of primary solid components. So it was confirmed that slag viscosity in highly basic slags ( $\text{C/S} > 1.3$ ) can be estimated by the chemical potential of dicalcium silicate.

They proposed through their studies that for a low value of  $\text{FeO}$  content of about less than 7.5% the slag viscosity showed minimum value with increasing  $\text{MnO}$  content. While, with the  $\text{FeO}$  content being more than that of 7.5% there is no particular effect on the slag viscosity with increasing  $\text{MnO}$  content. They also concluded that the BF slag viscosity decreases with increasing  $\text{FeO}$  content for a fixed  $\text{CaO/SiO}_2$  ratio. The variation in the slag basicity as well as the  $\text{Si}$  content in the metal can be minimized by less reduction of  $\text{SiO}_2$  into  $\text{Si}$ . This can be achieved by injection of flux in the blast furnace according to some tests conducted by some Japanese companies.



**Y.S.Lee, J.R.Kim, S.H.Yi and D.J.Min:** “Viscous behavior of CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>- MgO-FeO slag”. Proceedings of VIII international conference on molten slag, fluxes and salts, The South African Institute of Mining and Metallurgy, 2004, p.225.[5]

- Their studies showed that the heat transfer, mass transfer, SiO<sub>2</sub> and FeO reduction and gas permeability is controlled by the flow characteristics of the slag. This in turn plays an important role in the effect of the viscous nature of slag on the efficiency and productivity of the blast furnace. The viscous behaviour of CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO-FeO slags was studied under the conditions of CaO/SiO<sub>2</sub> = 1.15-1.6, 10-13 % Al<sub>2</sub>O<sub>3</sub>, 5-2% MgO, 5-20% FeO.
- Slag viscosity decreased with increasing slag basicity up to the CaO/SiO<sub>2</sub> ratio of 1.3.
- The silicon network repolymerizes and the silicate structure changes into simple chains of discrete anionic groups from the silicate 3D thus a decrease in basicity occurs.
- The FeO content in slag if increases the viscosity of the slag decreases so when the FeO decreases from 10-15% so the viscosity of slag at basicity of CaO/SiO<sub>2</sub> = 1.5 increases from about 2.5 to 10 dPa.s with increase in FeO content as mentioned above.

The viscous behavior of CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO-FeO slag and measured the viscosity under conditions of C/S ratio 1.35-1.45, 10-18% Alumina, 3.5-10% MgO and 5% FeO. They basically studied the influence of MgO and Al<sub>2</sub>O<sub>3</sub> alteration on the viscosities of BF slag. For a fixed MgO content and basicity the viscosity increases with increase in the Alumina content. The slag also showed a minimum value of viscosity at around 7% of MgO at over the temperature of 1723K. The MgO content variation did not significantly change the slag viscosity.

**Amitabh Shankar et al [15]** for the CaO-SiO<sub>2</sub>-MgO-Al<sub>2</sub>O<sub>3</sub> and CaO-SiO<sub>2</sub>-MgO-Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> system by varying the C/S ratio between 0.72 and 1.23 in the temperature range of 1573-1873 K. Alumina content was varied between 21-28%, Magnesia was varied between 2-8% and Titania was varied between 0-2%. They have shown that viscosity decreases with increase in basicity. It was also shown that the slope of the Viscosity vs. Temperature curve is steeper for low basicity slags. An increase in basicity decreases the slag viscosity, because silicate structure changes from network to discrete anionic groups containing simple chains or rings as basic oxides are increased.



**J.-Y. JIA, C.-G. BAI, G.-B. QIU, D.-F. CHEN and Y. XU [6]** a calculation model was established based on the studies of the ternary slag system of CaO-SiO<sub>2</sub>-TiO<sub>2</sub>. Thus, they were able to form a mass action concentration calculation model and viscosity calculation model based upon the existing theories and documented data at different temperatures of the ternary slag system at different compositions. The results obtained from the calculation are consistent with the literature values.

- With increasing TiO<sub>2</sub> content the mass action concentration also increases. This is also applicable practically.
- Viscosity of slag decreases with increasing TiO<sub>2</sub> content in the slag.
- Temperature is a key to viscosity. If the temperature rises, viscosity decreases, and running quality is good.

## **2.7. Effect of MgO and Al<sub>2</sub>O<sub>3</sub> content on viscosity of the slag**

**Seong-Ho Seok et al [17]** the viscous behavior of CaO-SiO<sub>2</sub>-FeO-MgO and CaO-SiO<sub>2</sub>-FeO-Al<sub>2</sub>O<sub>3</sub>-MgO melts were studied which were saturated with dicalcium silicate with a MgO content of 8% under conditions of high basicity and temperature of around 1873K. Through their studies they inferred that the viscosities of slag depend relatively more on the alumina content as the solid phases present is more in case of alteration in alumina content than that of MgO.

**Yasuji Kawai [8]** studied on the viscosities of molten slags and on the viscosities of CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO slags. When MgO was added to CaO-SiO<sub>2</sub> slags, the viscosities decreases with increasing amount of MgO up to about 20%, beyond which, however, it increased. The region of low viscosity was greater than that in CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> slag.

**Noritaka Saito et al [26]** studied the effect of MgO on the viscosity of 40CaO-40SiO<sub>2</sub>-20Al<sub>2</sub>O<sub>3</sub> slags. They suggested through their studies that magnesia acts a network modifier and also that the activation energy for viscous flow also decreases with addition of MgO. And they were able to show through their experiments that the viscosity of the slag decreases with that of addition of MgO.

**Masashi Nakamoto et al [16]** they used the rotating cylinder method to measure the viscous behavior of molten  $\text{CaO-SiO}_2\text{-MgO-Al}_2\text{O}_3$  and compared the result with its model that was created. They actually wanted to study the viscosity of slags that melt at low temperatures to improve the blast furnace operations at lower temperatures i.e. at around 1673K. They showed that slag of the following composition 35%  $\text{Al}_2\text{O}_3$ -43.1%  $\text{CaO}$ -7.5%  $\text{MgO}$ -14.4%  $\text{SiO}_2$  has a viscosity less than 0.6 Pa.s below 1673 K and it melts at around 1673K and below.

## **2.8. Effect of slag basicity on the liquidus temperature of the slag**

**R.K. Verma et al [7]** studied the effect of basicity on the solidus and liquidus temperature of the blast furnace type slags. They have found that, in general, liquidus and solidus temperatures are high for higher basicity slags. They studied the liquidus and solidus temperatures of the slags in the basicity range of 0.8-1.7. From the results obtained, it was suggested that basicity of around 1.05 is practicable in Indian blast furnaces.

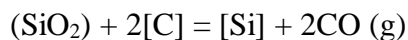
## **2.9. Cohesive zone model of blast furnace**

**S.Z. Li, Jian Cheng, and W.Q. Ma's [29]** the quality of iron and the efficiency of the blast furnace too much depends on the cohesive zone properties of the blast furnace. Thus they described a method to predict the shape of the cohesive zone of the blast furnace. This model developed can be observed during the operation of the blast furnace. This model is based on the principle of the conservation of the heat and mass transfer which is abided by the chemical reactions between coke, ore and the gas. Following which the blast furnace is divided into a series of homocentric circles and in vertical direction and each equation is resolved on each of the homocentric circles depending upon various parameters. The relationship between the temperature and the height thus can be established. Thus the cohesive zone can now be predicted based on the fusibility character of the ore. Thus this process is applied to real blast furnace and the results related to cohesive zone properties obtained are observed and evaluated.

## **2.10. Effect of silicon and its oxides on properties of slag and metal**

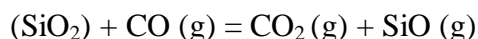
**B.Ozturk and R.J.Fruehan:** "Kinetics of the Reaction of  $\text{SiO (g)}$  with carbon saturated Iron": Metall.Trans.B. Vol.16B, 1985, p.121 [27]

Silicon transfer is considered to be very important in the iron blast furnace and this concern is explicitly explored in this paper. The main reaction which takes place in blast furnace for the Silicon transfer is given as follows:-

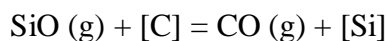


When coke reacts with its ash in the tuyers zone, there's formation of SiO because there's presence of the silicon element in the charged coke. The reaction of silicon with carbon is critical for silicon transfer in iron blast furnace. When carbon saturated iron droplets passes through the furnace, it reacts with the SiO (g) to put Si into the metal.

At the slag-gas interface CO reduced the SiO<sub>2</sub> to SiO (g)



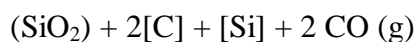
At metal-gas interface SiO reacts with dissolved carbon



CO<sub>2</sub> oxidizes carbon to form CO



So, the overall reaction taking place is summarized to,



The reaction of carbon with SiO is the rate controlling mechanism which can be controlled by the following:

- Gas phase mass transfer of SiO (g) to the metal surface.
- Diffusion of the reactant in liquid phase (carbon) to the surface or product (Silicon) into the metal.
- Chemical kinetics on the metal surface.
- Transfer of the product CO in gas phase away from the surface.

**B.Ozturk and R.J.Fruehan:** "The Reaction of SiO (g) with liquid slags": Metall.Trans.B. Vol.17B, 1986, p.397. [28]

The rate of transfer of silicon is too slow which is based on the results obtained from the slag-metal reaction so actual silicon content in the blast furnace is difficult to obtain. But now alternative mechanisms are available to the slag-metal reaction for which SiO gas is used. This paper thus indulges in the discussion of transfer of Silicon in the blast furnace.

The carbon saturated iron droplets reacts with the SiO generated as well as with the slag droplets. The gas phase mass transfer controls the kinetics of reaction between SiO in gaseous form and carbon dissolved in.

# CHAPTER-3

---

## EXPERIMENTAL DETAILS

## CHAPTER-3 EXPERIMENTAL WORK

### 3. Experimental Details: Theory and Procedure

#### 3.1. Flow Characteristics of Blast Furnace Slag

High temperature microscope is used to determine flow characteristics of slag sample. It has got four characteristics temperatures to be studied:

- Initial deformation temperature (IDT)
- Softening temperature (ST)
- Hemispherical temperature (HT)
- Flow temperature (FT)

The followings are defined as per German Industrial Standards 51730. [18]

##### 3.1.1. Initial Deformation Temperature (IDT)

Initial deformation temperature is the temperature at which the first rounding up of the edges of the cube-shaped sample specimen takes place. In fact this is the temperature at which the first sign of the change in shape appears. Rheologically this temperature symbolizes the surface stickiness of the slag.

##### 3.1.2. Softening Temperature (ST)

It is the temperature at which the outline of the shape of the sample starts changing and is reported as the temperature at which the sample shrinks by one division or the temperature at which the distortion of the sample starts. Rheologically this temp symbolizes the start of plastic distortion.

##### 3.1.3. Hemispherical Temperature (HT)

It is the temperature at which the sample has fused down to hemispherical shape and is measured as the temp at which the height of the sample is equal to the half of its base length. This is defined as the fusion point or the melting point in **German Industrial Standards 51730**. Rheologically this temperature symbolizes the sluggish flow of the slag.

##### 3.1.4. Flow Temperature (FT)

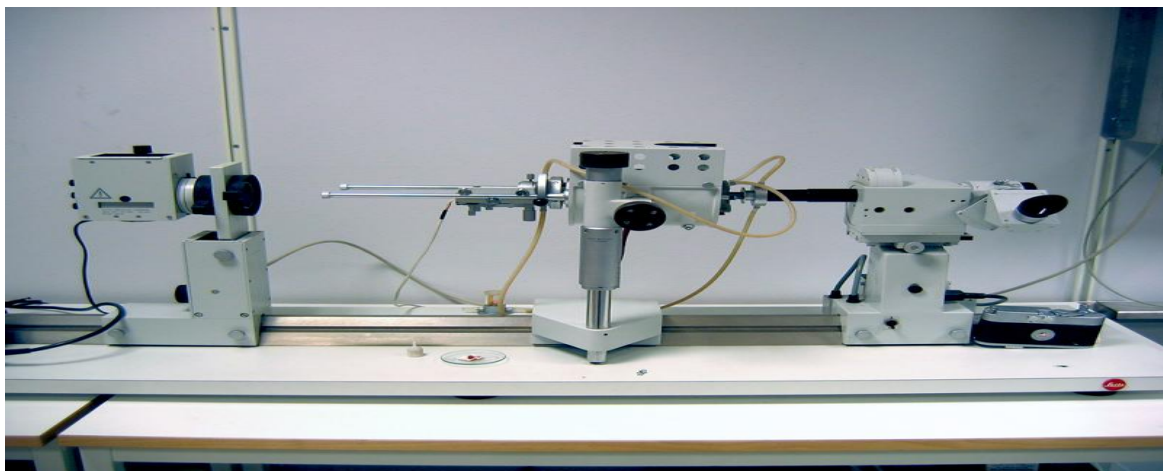
It is the temperature at which the sample liquidities and is reported as the temperature at which the height of the sample is equal to one-third of the height that it had at HT

(hemispherical temperature). Though some books reported as the temperature at which the height becomes one third of the initial height. The former is more accurate and is universally accepted. Rheologically this temperature symbolizes the liquid mobility of the slag.

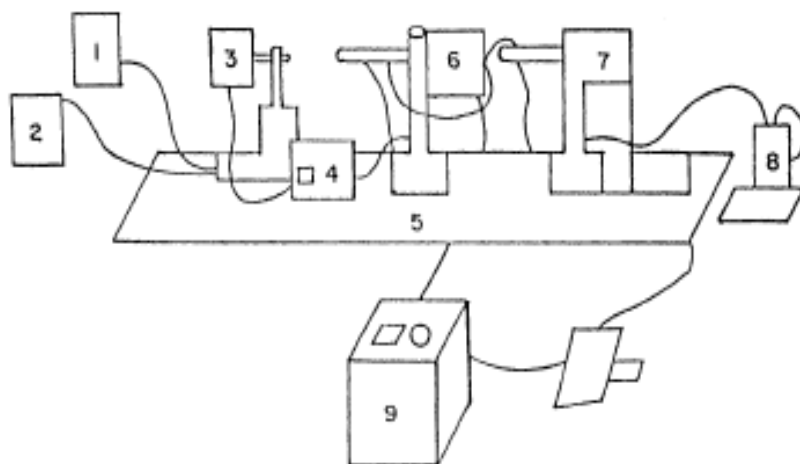
### 3.2. Experimental Apparatus

#### 3.2.1. High Temperature Microscope

The Heating Microscope method is adopted for recording the characteristic temperatures. A picture of the Leitz heating microscope is shown in Fig. 5. Schematic diagram of the instrument is presented in Fig. 6. The sample, in the form of a 3 mm cube, is heated in an electric furnace in the microscope assembly. The shape change of the sample as a result of heating is photographed by a camera. A grid-division which is simultaneously photographed with the sample and the temperature to which the sample is being heated facilitate identification of the four characteristic temperatures.



*Fig 5. Pictorial view of Leitz heating microscope*



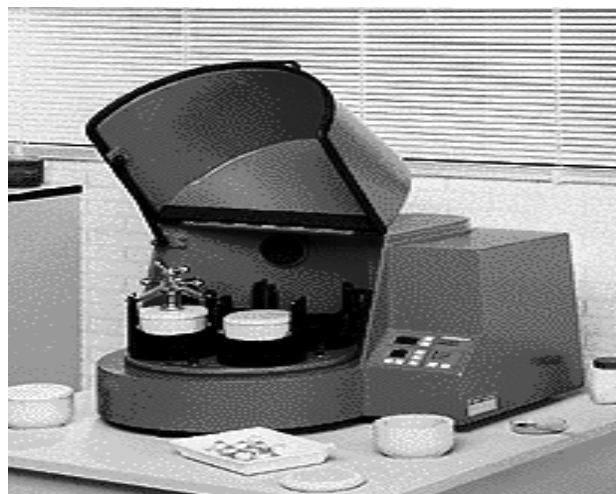
*Fig. 6 Schematic diagram of the heating microscope*

1. Cooling water tank
2. Cooling water recirculating tank
3. Light source
4. Regulating transformer for light source
5. Optical bench
6. High temperature electrical furnace with specimen carriage
7. Observation and photo microscope
8. Digital thermometer
9. Regulating transformer for high temperature electrical furnace

### 3.2.2 Planetary Ball Mill

These mills are also referred as centrifugal mills and are used to grind samples into colloidal fineness by generating high grinding energy.

Fig. 7. represents a four station planetary mill presented by Gilson Company. The samples are placed in one of the vile and numerous balls are added as shown. The vile is covered by the cover plate and then it is mounted in the machine. Once the viles are mounted and secured, the machine is functional. The bowls are independent of the rotatable platform and the direction of rotation of the bowls is opposite to the direction of the rotatable platform. The motion resembles the teacup and saucer as seen in some of the amusement parks.



*Fig. 7- a four station Planetary Ball Mill*

Due to alternate addition and subtraction of the centrifugal forces, the grinding balls rolls halfway in the vile and then thrown across the viles and then impacting the opposite walls at



very high speeds. 20g acceleration is reached due to the planetary action and the time for the grinding reduces about 2/3 times than a simple centrifugal mill.

### 3.2.3. Abrasion Tester Mixer

The Abrasion Testing mixer is used to mix the slag samples. A fixed 25 grams of total weight (containing weighted percentage composition of all the major oxides) of the combination of major oxides are taken into a plastic container. There are 3 such containers that can be put together, making it 75 grams. Since the mixing should be homogenous and perfectly mixed, each container of 25 g is separately taken weights as per calculations.



*Figure 8: Abrasion Testing Mixer*

This plastic container is then placed in one of the 3 rotating chambers of the mixer. A total of one lakh revolutions are required for appropriate mixing which is completed in 6 hours. 3 such sessions are required for complete mixing of 75 grams. So the total 145 gram of the required synthetic slag sample is prepared in 6 days. The mixing is very homogenous when done by this process and gives us thoroughly mixed oxides.

### 3.2.4. Sintering Furnace

The pellets obtained are sintered at around 1680 degrees Celsius in the sintering furnace to bind the slag material by the process of diffusion. This temperature is selected as it is the temperature that the slag undergoes when comes out of the blast furnace, so firing is done at this temperature. Proper firing can be easily done when homogenous mixing is done. There are two crucible holders where two platinum crucibles are places each containing different



slags. Thus at a time 2 slags can be prepared. After attaining the required temperature gradually, the slag is then quenched in water to room temperature with the crucible in different containers.



*Figure-9: Sintering Furnace*

After firing and quenching is done, the crucibles used are then cleaned using dilute HCl (50% water and 50% Conc. HCl). The cleaning process generally takes 2 days after which gel formation takes place for the slags that were stuck on the walls of the crucible. Platinum crucibles are used for the process as very high temperature is reached during firing and platinum can easily sustain that temperature and also is very nonreactive with the slags or the outer ambience.

### **3.2.5. Pelletizer Machine**

The total slag obtained is pelletized in the compression machine under 5 ton loads, where the slag is compacted between the dies under pressure. Thus small cylindrical pellets are formed with each pellet having almost the same dimension. The process of pelletization is done as the platinum crucible used for the firing of the oxides, are very small and cannot contain

powdered slag so making into pellets they become compacted and will take less space and so can be easily put in the crucibles for the firing procedure.



*Figure 10: Pelletizer Machine*

### 3.2.6. X-ray diffraction (XRD)

X- ray diffraction technique was used to identify the different phases (elemental phase/intermetallic phase/crystalline phase/non-crystalline phase) present in the coating. XRD analysis was done by using X-pert MPD system (PAN Analytical). Here Ni- filtered Cu-  $K\alpha$  radiation used in X- Ray diffractometer. d- Values obtained from XRD patterns were compared with the characteristics d-spacing of all possible values from JCPDS cards to obtain the various X-ray peaks. Obtained d-spacing based on the equation:

$$n\lambda = 2d\sin\theta$$

Where,  $\lambda$ = Wavelength of characteristic x-rays

d=Lattice interplanar spacing of the crystal

&  $\theta$ =x-ray incident angle



*Figure 11: X-ray Diffraction Machine*

### **3.2.7. Scanning electron microscopic Machine**

By using JEOL JSM-6480 LV scanning electron microscope (SEM), microstructure of raw power and plasma sprayed coated specimens were studied. The surface morphology as well as the coating – substrate interface morphology of all coatings was observed under the microscope. Here SEM mostly using the secondary electron imaging.

### **3.3. Experimental Work**

The experimental work is planned in ‘THREE’ phases, the aim being to determine the characteristics temperatures of the B.F. slag for proposing a composition that would indicate narrowed down. The three phases of experimentation include the following:

1. Collecting B.F. slag from different industrial Blast furnaces; study of the flow characteristics and chemical analysis to determine the most desired composition (to ensure a narrow cohesive zone lower down the furnace).
2. To Prepare synthetic slag in the laboratory with pure oxides as obtained from the market, resembling the chemical composition of the slag with best results (narrow cohesive zone lower down the furnace) as per the first set of experiments determination of the flow characteristics of these synthetic slags and

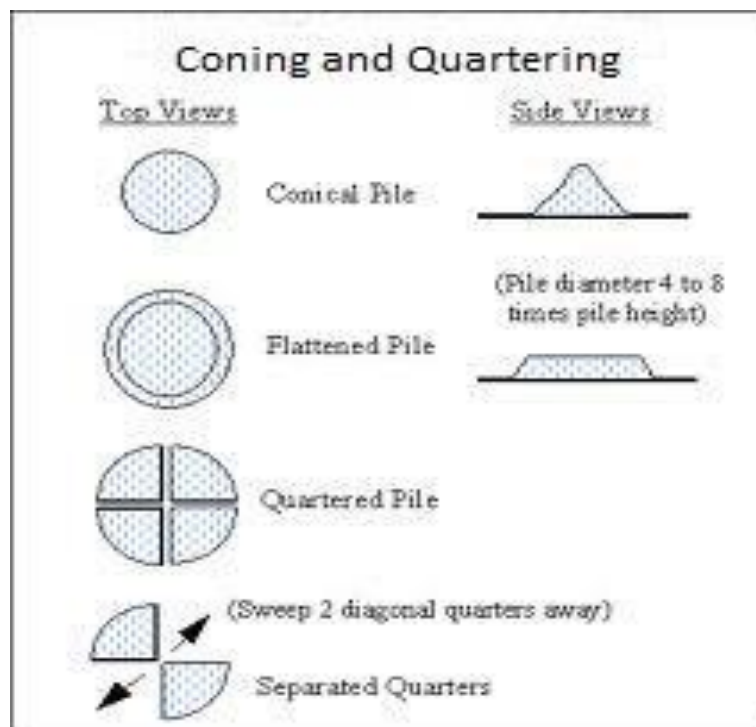
3. (a) Preparation of pellets of Hemetite varying the composition around the same as obtained from the first set of experiments.
- (b) Determination of Tumbler Index and Abrasion Index of cold-set pellets
- (c) Subjecting the pellets to different experts of reduction and
- (d) Determination of the flow characteristics of the reduced pellets.
- (e) XRD analysis of these pellets to determine the different phases.
- (f) SEM analysis to determine the microstructure.

### 3.4. Experimental Procedure

#### Preparation of samples (Blast Furnace slag from industry)

##### **Sampling:**

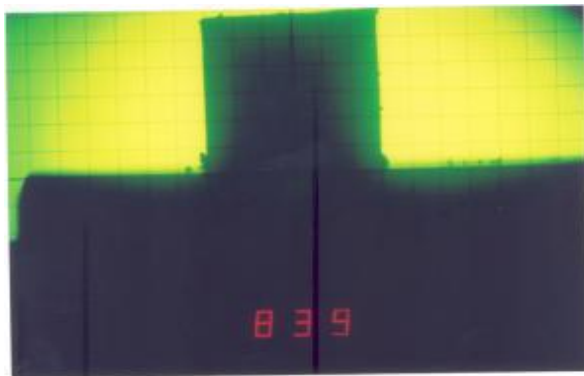
Sampling is usually done and especially for the case of coning and quartering for the purpose of checking the uniformity and the homogeneity of the sample prepared. For the method of coning and quartering the sample is first powdered and then laid out on a clean surface in the form of a conical heap. The cone is then quarters into four parts, following which only two opposite quarters are considered (like first and third) while the other quarters (second and fourth ) are discarded. the samples are taken from each of the two opposite quarters and those small quantity of sample are formed into small cubes .The process is repeated as many times as necessary to obtain the quantity desired for some final use. [14]



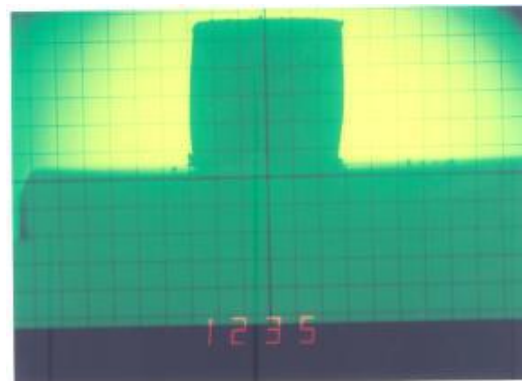
**Figure 12: Coning and Quartering**

**Analysing:**

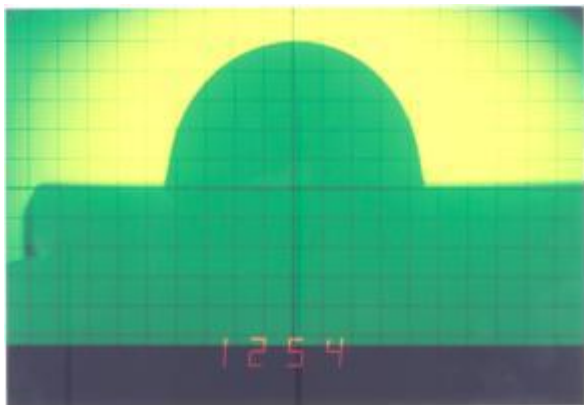
Analysis part consists of the high temperature microscopy. Leitz heating microscope is used for this process. The powdered slag is prepared in the form of small cubic shapes for testing. They are mounted in the heating microscope. The sample gets heated gradually and deformation takes place. This deformation defines the flow characteristics of the slag in the form of IDT, ST, HT and FT. There is a control of heating rate, water is used as coolant and there is a camera attached to take photographs of different characteristics temperature of slags when required.



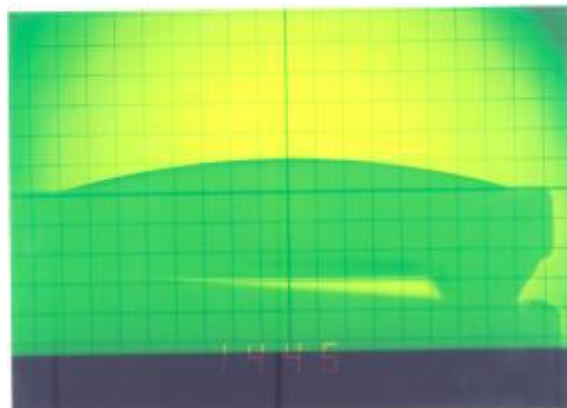
Initial Deformation Temperature



Softening Temperature



Hemispherical Temperature



Flow Temperature

**Figure 13:** View of Different Flow characteristics of the slag prepared in the lab observed through Leitz Heating Microscope

**Preparation of samples (synthetic slag)**

Slags from different blast furnace of different plants such as Rourkela Steel Plant (RSP) and TATA Steel were analysed to get a base idea of the expected synthetic slag composition and thus an average slag composition was determined. An Average Slag is a slag arrived by

taking the average of the maximum and minimum composition that we got from the slags collected from different blast furnaces.

Five different basicity ratio ( $\text{CaO}/\text{SiO}_2$  ratio) or (c/s ratio) were obtained by some variation in C/S ratio of the average slag that was determined. Also five different MgO and  $\text{Al}_2\text{O}_3$  variation were included. Thus different combinations of the synthetic slag are possible for these particular combinations.

For our experimentation we first took a particular constant value of MgO and only C/S ratio was varied. Next some slags were prepared by taking the Basicity ratio as constant and varying the MgO compositions. Thus various combinations can be prepared and then analysed to get the best slag. Slag compositions obtained now needs to be prepared from the major oxides such as the CaO, MgO,  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ . The major oxides are obtained individually from the commercial market which are about 99% pure.

The following is the detailed sample preparation list.

### **Sample Preparation:**

We are making 150 g of slag sample. Minor constituents taken = 3.66 % i.e. nearly 5 g in 150 g of total slag. This includes  $\text{TiO}_2$ , FeO. We have neglected the minor slags in our experiment. For the sample preparation after obtaining almost 99% pure oxides of CaO, MgO,  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ , following processes are involved.

### **Different Process Involved:**

#### **A) Furnace Heating:**

CaO and MgO content are heated at 200 degrees Celsius to remove moisture. Furnace heating at 200 takes usually 1-2 hours of time and is very effective in removing moisture which can increase the weight of the oxides and thus create a problem.

#### **B) Weighing:**

After the oxides are furnace heated, they are then weighed and mixed according to their percentage composition in the digital weighing machine accurately.

#### **C) Mixing:**

After taking proper accurate weights of the oxides they need to be thoroughly mixed. The mixing of the major oxides is done in the abrasion tester machine mixer. A fixed 25 grams of total weight (containing weighted percentage composition of all the major oxides) of the combination of major oxides are taken into a plastic container. There are 3 such containers



that can be put together, making it 75 grams. Since the mixing should be homogenous and perfectly mixed, each container of 25 g is separately taken weights as per calculations.

This plastic container is then placed in one of the 3 rotating chambers of the mixer. A total of one lakh revolutions are required for appropriate mixing which is completed in 6 hours. 3 such sessions are required for complete mixing of 75 grams. So the total 145 gram of the required synthetic slag sample is prepared in 6 days. The mixing is very homogenous when done by this process and gives us thoroughly mixed oxides.

#### **D) Pelletization:**

The total slag then obtained is pelletized in the compression machine under 5 ton load, where the slag is compacted between the dies under pressure. Thus small cylindrical pellets are formed with each pellet having almost the same dimension. The process of Pelletization is done as the platinum crucible used for the firing of the oxides, are very small and cannot contain powdered slag so making into pellets they become compacted and will take less space and so can be easily put in the crucibles for the firing procedure.

The pelletization process generally takes 3 hours continuously for converting the 150 g of sample powder into form of pellets. After the formation of pellets it must be kept away from moisture and must be used for the firing process as soon as possible else the pellets can break back to powder form in due time.

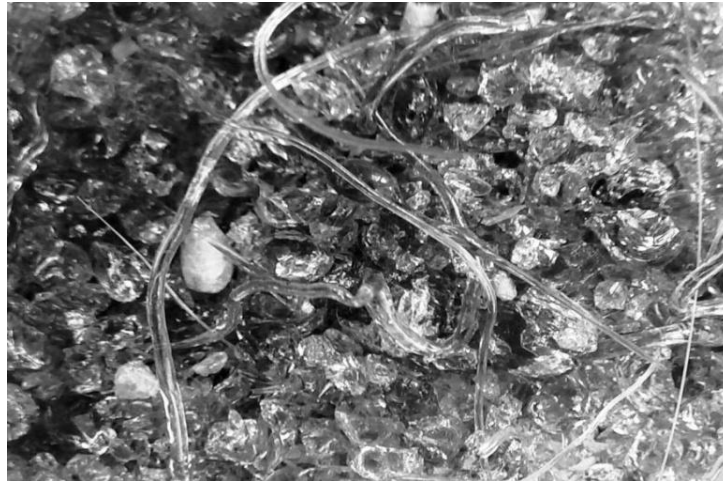
#### **E) Firing:**

The pellets thus obtained are then sintered at around 1680 degrees Celsius in the sintering furnace to bind the slag material by the process of diffusion. This temperature is selected as it is the temperature that the slag undergoes when comes out of the blast furnace, so firing is done at this temperature. Proper firing can be easily done when homogenous mixing is done. There are two crucible holders where two platinum crucibles are placed each containing different slags. Thus at a time 2 slags can be prepared. After attaining the required temperature gradually, the slag is then quenched in water to room temperature with the crucible in different containers.

#### **F) After Firing:**

After firing and quenching is done, the slags are taken out from the crucibles and heated to remove the water content. They are kept in the furnace at a temperature of 150 degrees Celsius for 1 hour. The crucibles used are then cleaned using dilute HCl (50% water and 50% Conc. HCl). The cleaning process generally takes 2 days after which gel formation takes place for the slags that were stuck on the walls of the crucible. Platinum crucibles are used for

the process as very high temperature is reached during firing and platinum can easily sustain that temperature and also is very nonreactive with the slags or the outer ambience.



*Figure 14: Synthetic slag prepared in the laboratory*

#### **G) Ball Milling:**

The sintered slag pellets after furnace heating are then reduced to fine powder by the process of ball milling in the planetary ball mill. This process usually requires about 30 minutes. 300 rpm is used for the milling process. After this the slag is obtained as fine powder from the planetary mill. Thus the mechanical and physical process for the sample preparation concludes with this process.

#### **H) Sample Analysis:**

The procedure is same as performed in the analysis of industrial slag method.

#### **Preparation of pellets of Hemetite varying the composition around the same as obtained from the first set and second set of experiments.**

The compositions of green pellets are as follow:

Fe<sub>2</sub>O<sub>3</sub> – 63.66 %

SiO<sub>2</sub> – 5.46 %

CaO – 5.54 %

MgO – 11.4 %

Al<sub>2</sub>O<sub>3</sub> – 13.5 %

The 2% of bentonite is used as a binder then we heated the green pellet at 950<sup>0</sup>C for 2 hour then we took iron crucible a bed of 20 mm reductant was fed in to the crucible before pellets kept inside the crucible some more reductant were added to cover the pellets followed by



layer of fired clay, to avoid air oxidizing reductant. Then the crucible was placed in the furnace the temperature of furnace was kept constant  $1100^{\circ}\text{C}$ , then the first pellet was withdrawn after one hour, next pellet was withdrawn after two hour and the last pellet was withdrawn after three hour and % of reduction was calculated for each pellets.

$$\% \text{ Reduction} = (\text{loss in weight} / \text{theoretically attached Oxygen}) \times 100$$

For example, Initial weight of iron ore is x gram

Then 100 gram of Iron ore containing Fe is 64 gms

Therefore for the weight of x gms of Iron ore =  $(64/100) \times x$  of iron is present

And we know that (for 1 mole of  $\text{Fe}_2\text{O}_3$ )

Since, 112 gms Fe is associated with 48 gms of Oxygen

Therefore  $(64/100) \times x$  gms of Fe is associated with =

$$(48/112) \times (64/100) \times x \text{ gms of Oxygen}$$

That means the theoretical attached oxygen in  $(64/100) \times x$  gms of Fe is

$$[(48/112) \times \{(64/100) \times x\}] \text{ gms of } \text{O}_2$$

### **Determination of Tumbler Index and Abrasion Index of cold-set pellets**

The test was carried out in the Drum-Tumbler index testing equipment having 1 meter length and 0.8 meter diameter. 15 to 20 kg of 9 to 16 mm pellets were chosen for the test. The pellets were put in the drum and it was rotated of 25 rpm for 200 revolutions.

The total charge was then collected and made to pass through

- (i) 6 mm sieve and
- (ii) 0.25 mm sieve.

Tumbler Index – The fraction collected over the 6 mm screen (+6 mm) was reported as tumbler index. It was found to be 94.

Abrasion Index – The fraction passing through the 0.25 mm screen (-0.25 mm fraction) was reported as tumbler index. The Abrasion index was found to be 0.6.

The above tests indicated that the pellets would stand the harsh environment in the blast furnace. Keeping the bed open and are separated for use in a blast furnace.

### 3.5. Experimental Result and Discussion

#### 1) PHASE- I INDUSTRIAL SLAGS

Table (1) below gives the compositional details of the slags obtained from various blast furnaces in the country

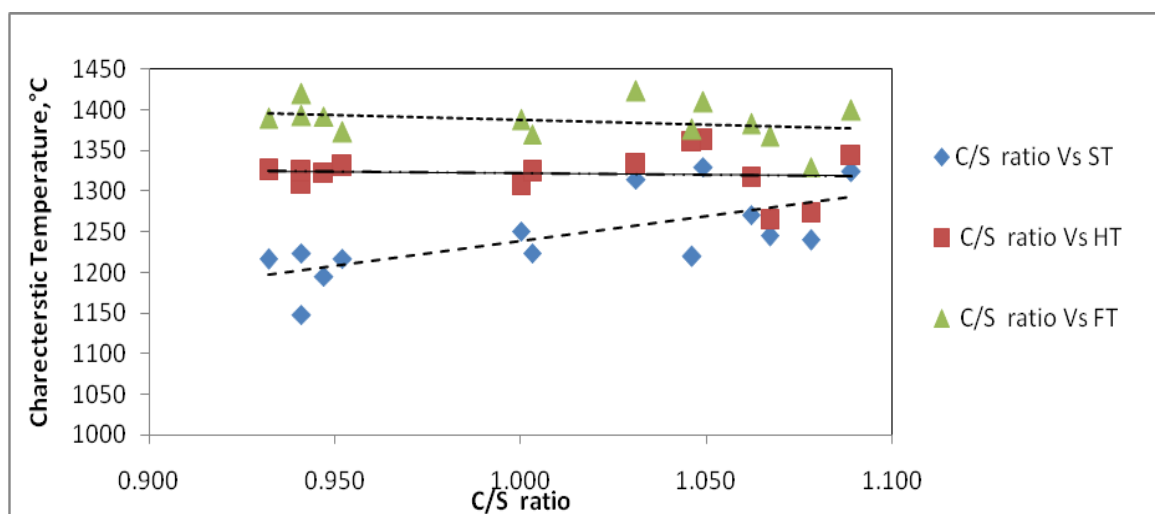
*Table (1): Chemical Composition of Industrial Blast Furnace Slags*

Serial No	CaO %	SiO <sub>2</sub> %	MgO%	Al <sub>2</sub> O <sub>3</sub> %	C/S	Remarks
1	34.04	32.05	10.09	19	1.062	The slag samples are collected from SAIL Rourkela; TATA Steel plant; Bhilai Steel plant and Bokaro Steel plant
2	33.25	30.84	11.01	20.89	1.078	
3	35.1	32.24	10.4	18.77	1.089	
4	33.4	31.3	10.4	19	1.067	
5	32.55	31.58	10.4	20.28	1.031	
6	33.61	32.05	10.09	20.05	1.049	
7	30.85	30.85	9.79	18.54	1.000	
8	36.2	34.6	7.05	17.9	1.046	
9	34.57	36.72	6.51	19.04	0.941	
10	34.15	34.06	6.5	18.07	1.003	
11	31.9	33.5	10.4	20.8	0.952	
12	31.9	33.7	10.5	20.6	0.947	
13	31.6	33.9	10.6	20.6	0.932	
14	31.8	33.8	10.5	20.6	0.941	

TABLE II below gives the slag-wise characteristics temperatures.

*Table II: Characteristics Temperature of Industrial Blast Furnace Slag*

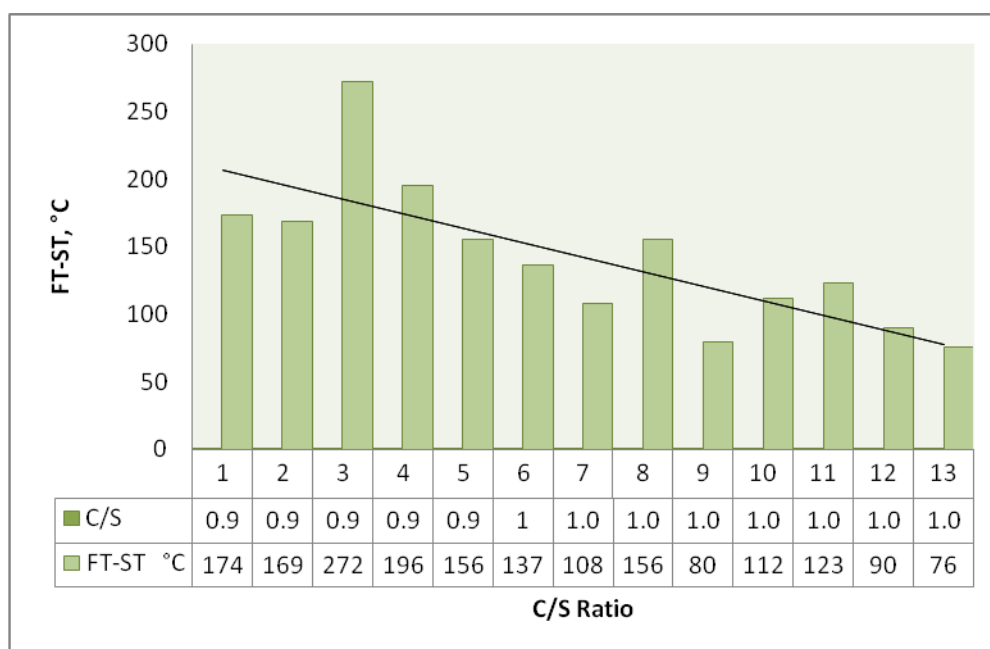
Serial No	IDT °C	ST °C	HT °C	FT °C	FT-ST °C
1	1203	1271	1318	1383	112
2	1220	1240	1274	1330	90
3	1210	1324	1345	1400	76
4	1204	1245	1266	1368	123
5	1225	1315	1335	1423	108
6	1217	1330	1363	1410	80
7	1200	1251	1307	1388	137
8	920	1220	1362	1376	156
9	827	1148	1310	1420	272
10	810	1224	1324	1370	146
11	817	1217	1331	1373	156
12	820	1195	1323	1392	196
13	813	1216	1326	1390	174
14	818	1224	1324	1393	169



**Figure.15.** Variation of different characteristic temperatures with C/S ratio

The experimental data is plotted in fig no.15. As evident from the figure it is seen that the softening temperature of the industrial slags increase with the c/s ( $\text{CaO}/\text{SiO}_2$ ) ratio. It can also be seen that the flow temperature at which the slag acquires liquid mobility, decreases with the c/s ratio and in general the rate of decrease of the flow temperature is low and low as the c/s ratio goes up.

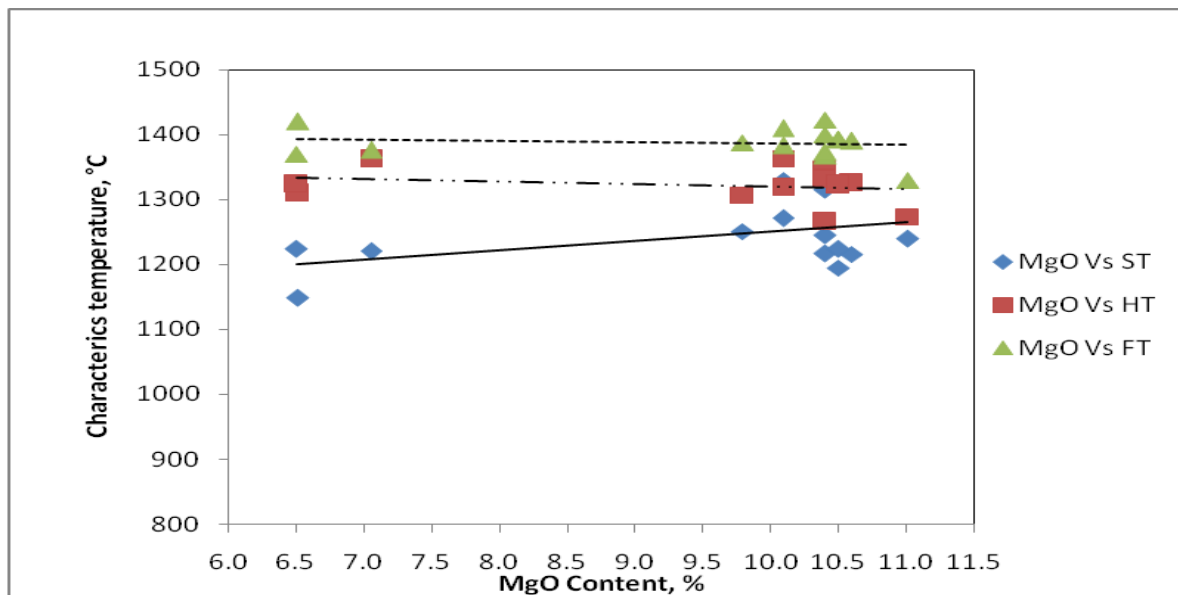
U.K.Mohanty [12] observed that with the addition of the basic oxide CaO the silicate network gets depolymerised. The result is the formation of smaller silicate groups or anionic units or flow units. We know that with the formation of smaller anionic groups the activation energy of the melt is lowered and the viscosity is reduced. Here the decrease of flow temperature may be considered as analysis to the decrease in viscosity. It is further known that smaller and smaller flow units, associated with lesser and lesser energy of viscous flow need relatively high oxygen which come from the higher additions of basic oxide. Thus progressive increases in the basic oxide content is less and less effective in causing depolarization bringing down the flow temperature the slag low in viscosity acquiring higher liquid mobility. Thus the flow temperature decreases only at a decreased rate with increase in the c/s ratio [13].



**Figure.16.** Variation of (FT – ST) with C/S ratio

The net effect of increase in ST and relative decrease in FT establishes that the 0 gap 6 between ST and FT (FT-ST) decreases with increase of c/s ratio, in general, for the blast furnace slags. Thus, for the industrial slag, in general under the range of compositions

studied, as increases of c/s ratio result in lowering of the cohesive zone (high ST) and developing a narrow (small FT-ST) cohesive zone. This is evident from fig no-16 which plots the gap between the FT and the ST (FT-ST) as a function of the c/s ratio.



**Figure.17.** Variation of different characteristic temperatures with MgO content

Variation of the characteristic temperatures with variation of MgO content of the slag is presented in fig. no. 17.

The figure reveals that high MgO is also beneficial in lowering the cohesive zone and narrowing it down, as discussed above. It is further revealed that a high c/s ratio coupled with a high MgO value results in short-slugs with the difference between the FT and ST being less than 100°C. Such a slag, as soon as it distorts, flows down the furnace. These results in the higher rate of exposure of slag- metal interface and are certain to enhance the slag metal interaction, thus enhancing the slag metal reaction rates.

As evident from Table no 2, slag nos. 2, 3 and 6 are short-slugs. These slags have a c/s ratio varying between 1.049 to 1.089 and MgO between 10.09 to 11.10 percentages.

The 1<sup>st</sup> phase of experiments thus reveals that the range of comparison most suitable fall between c/s ratio values of 1.049 to 1.089 and MgO percentages of 10.09 to 11.10. This data is used to design the experiments in phase 2 and phase 3 set of experiments, where the c/s ratio and MgO percentages are varied around these base values up and down. With these variations a set of slags are synthetically prepared for phase 2 set of experiments and a set of pellets are prepared and subjected to different extents of reduction for the phase 3 set of experiments.

## **2) PHASE II SYNTHETIC SLAGS**

As discussed earlier the 1st set of experiments provided the basis for preparing the synthetic slags in the laboratory. These slags prepared from pure oxides as obtained from market are categorized under three headings. Table no.3, table no 4 and table no 5 show the comparison of the slags varied systematically. In these slags  $\text{Al}_2\text{O}_3$  is kept constant at 19.71%.

*Table no. 3 Variation of c/s ratio at fixed MgO percentage*

Sl. no.	c/s ratio	MgO%	CaO %	$\text{SiO}_2\%$	$\text{Al}_2\text{O}_3\%$
1	0.907	9.26	32.04	35.33	19.71
2	0.96	9.26	33	34.37	19.71
3	1.01	9.26	33.85	33.52	19.71
4	1.07	9.26	34.82	32.55	19.71
5	1.13	9.26	35.74	31.63	19.71

*Table no. 4 Variation of MgO percentage at fixed c/s ratio*

Sl. no.	c/s ratio	MgO%	CaO %	$\text{SiO}_2\%$	$\text{Al}_2\text{O}_3\%$
6	1.01	7.4	34.787	34.443	19.71
7	1.01	8.32	34.325	33.985	19.71
8	1.01	9.26	33.852	33.517	19.71
9	1.01	10.2	33.38	33.049	19.71
10	1.01	11.9	32.526	32.204	19.71

*Table no. 5 Variation of MgO (theoretical) at fixed c/s ratio*

Sl. no.	c/s ratio	MgO%	CaO %	$\text{SiO}_2\%$	$\text{Al}_2\text{O}_3\%$
11	1.1	4	38.04	34.59	19.71
12	1.1	6	36	33.63	19.71
13	1.1	8	35.95	32.68	19.71
14	1.1	10	34.9	31.73	19.71
15	1.1	12	33.85	30.78	19.71

The characteristic temperature for slags preparing to table no. '3', '4' and '5' are prepared in table no. 6 through '8'.

*Table 6: Characteristics Temperature of Slags presented in Table '3'*

Serial No	IDT °C	ST °C	HT °C	FT °C	FT-ST °C
1	813	1232	1248	1424	192
2	838	1232	1249	1430	198
3	836	1242	1250	1432	190
4	830	1232	1260	1435	203
5	826	1228	1275	1420	192

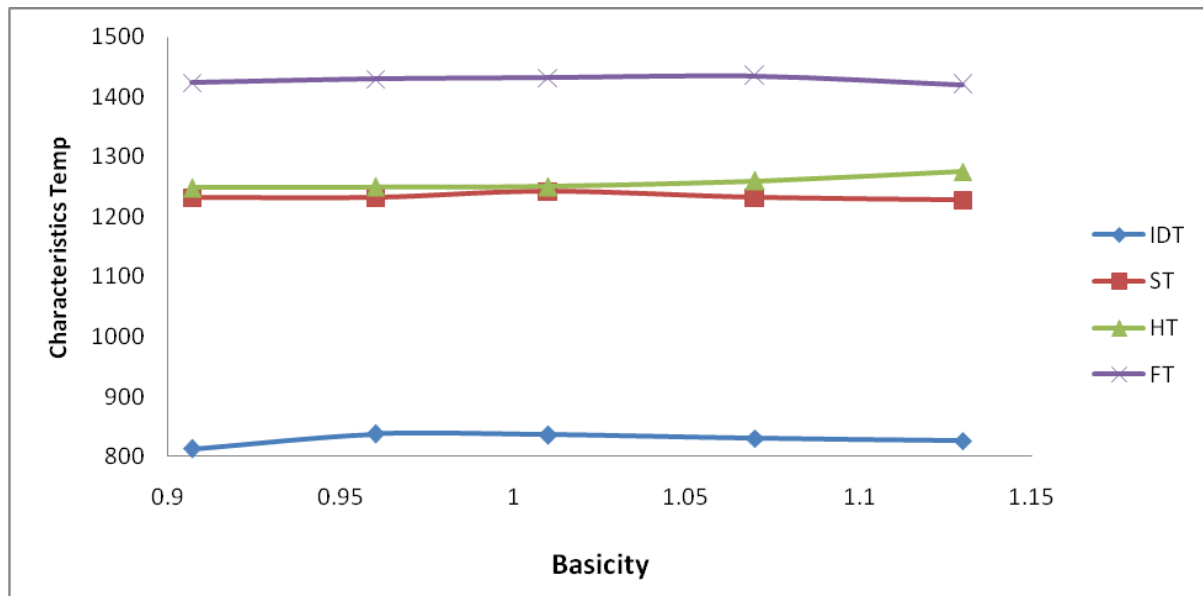
*Table 7: Characteristics Temperature of Slags presented in Table '4'*

Serial No	IDT °C	ST °C	HT °C	FT °C	FT-ST °C	Remark
6	821	1223	1253	1328	105	Slag no. 7 suffered from bubble formation till 1370°C and accurate measurement of FT was not possible
7	826	1235	1263	1332	97	
8	829	1236	1255	1345	109	
9	824	1232	1252	1342	110	
10	818	1224	1283	1353	129	

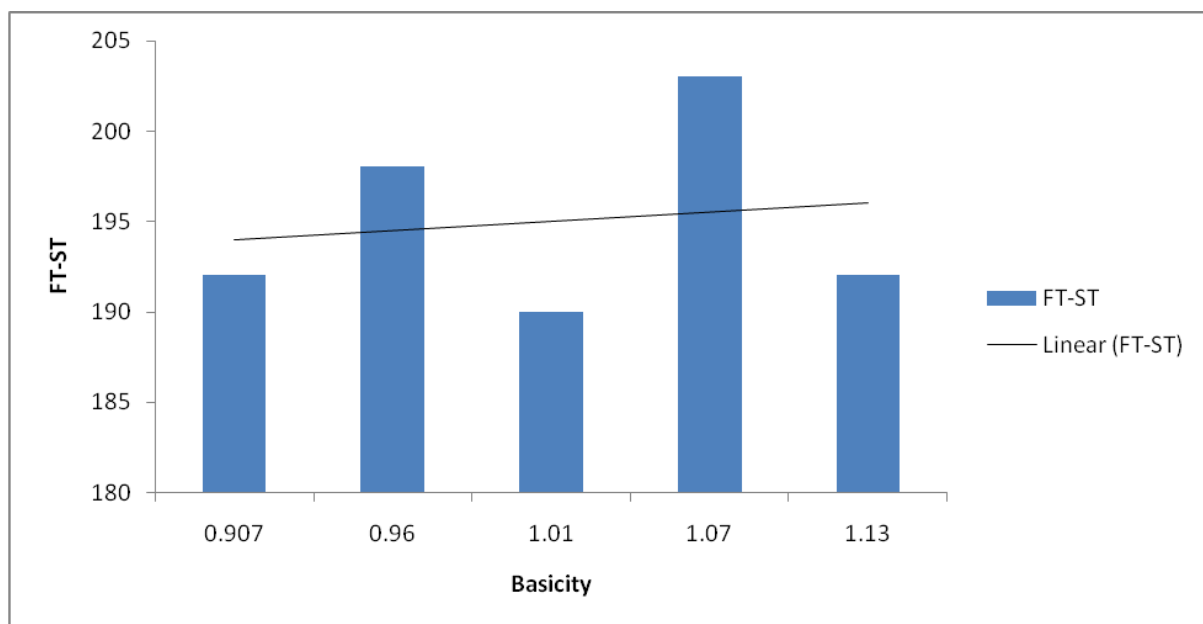
*Table 8: Characteristics Temperature of Slags presented in Table '5'*

Serial No	IDT °C	ST °C	HT °C	FT °C	FT-ST °C
6	832	1233	1285	1328	95
7	838	1270	1285	1365	95
8	838	1218	1260	1316	98
9	817	1227	1335	1440	213
10	815	1210	1300	1328	118

The data obtained is plotted in fig. no 18 through fig. no. 23 which shows the characteristic temperatures and the difference of FT and ST as a function of the c/s ratio and the MgO content respectively.



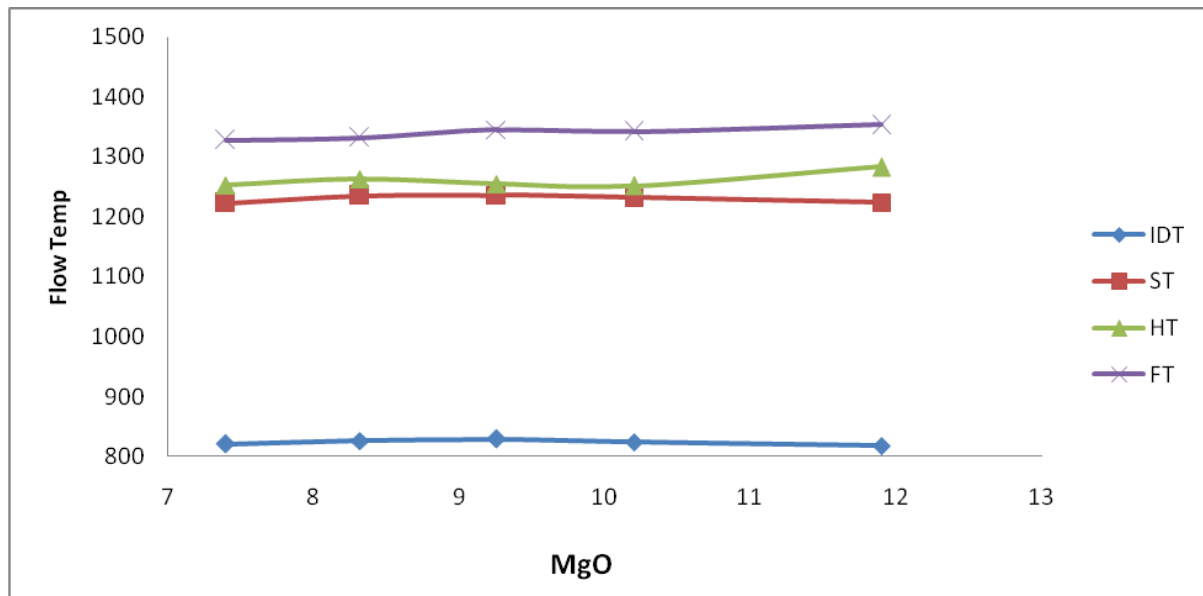
**Figure.18.** Variation of characteristic temperatures with C/S ratio for slag nos. 1-5



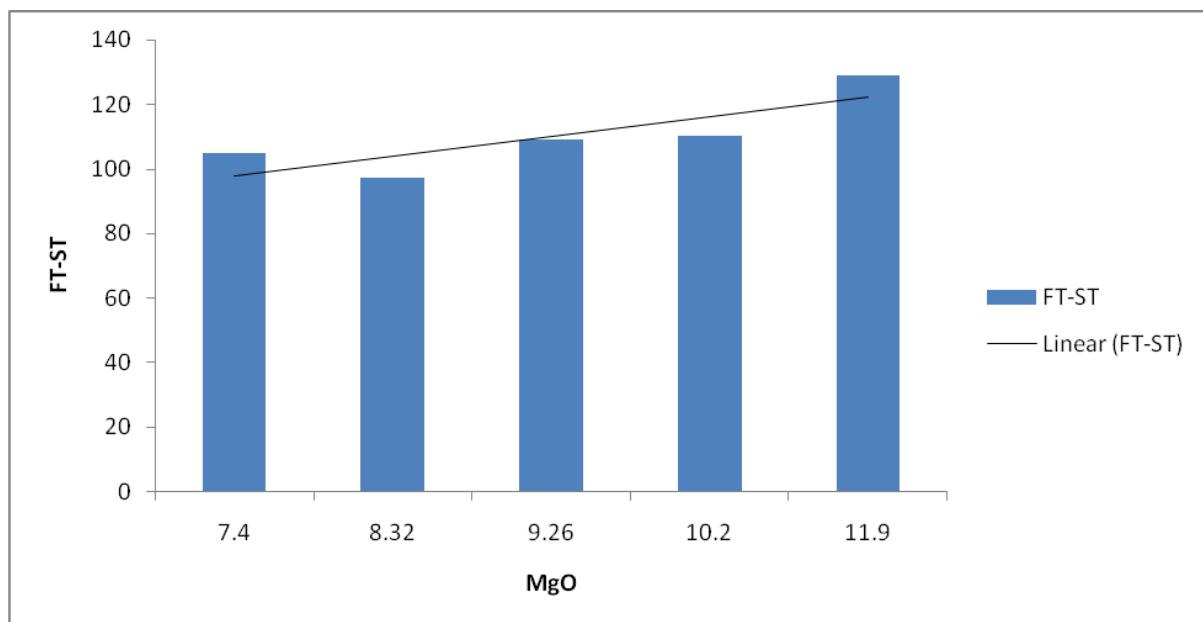
**Figure.19.** Variation of ST-FT temperatures with C/S ratio for slag nos. 1-5

Table no.6 and fig. no.18 reveal that MgO content at 9.26% with c/s ratio varying from 0.907 to 1.13 has no significant effect on the characteristic temperatures. In this category of slags studied, the difference between FT and ST is much above 100°C, meaning none of these slags are short-slugs. An increasing C/S ratio in these groups of slags exhibits an increase in difference between the FT and the HT through the difference is very nominal.





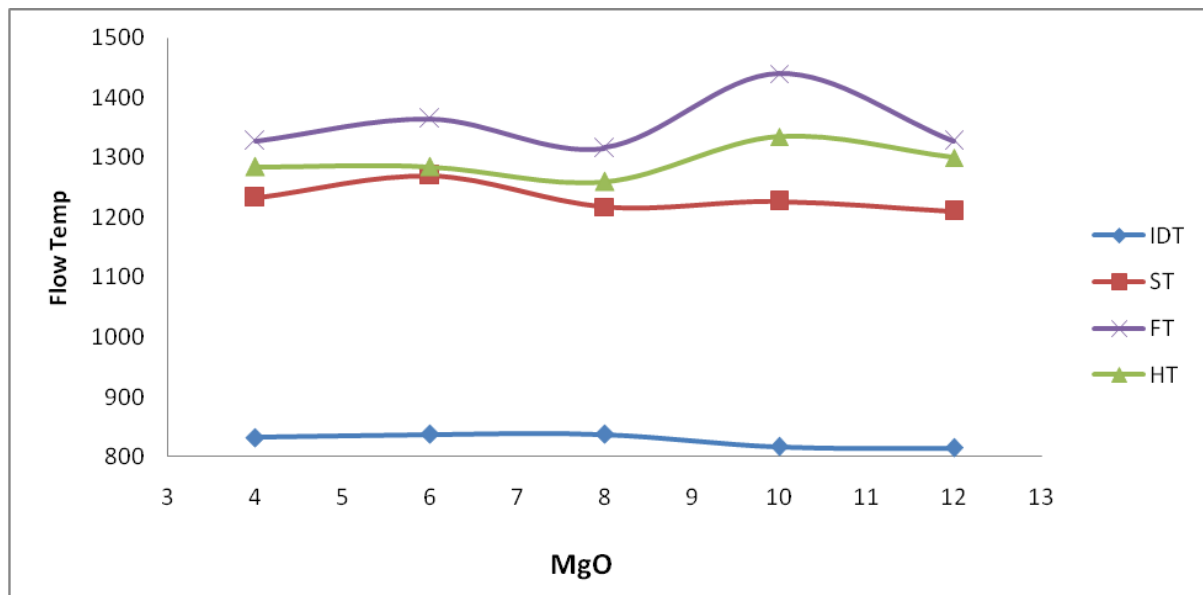
**Figure.20.** Variation of characteristic temperatures with MgO content for slag nos. 6-10



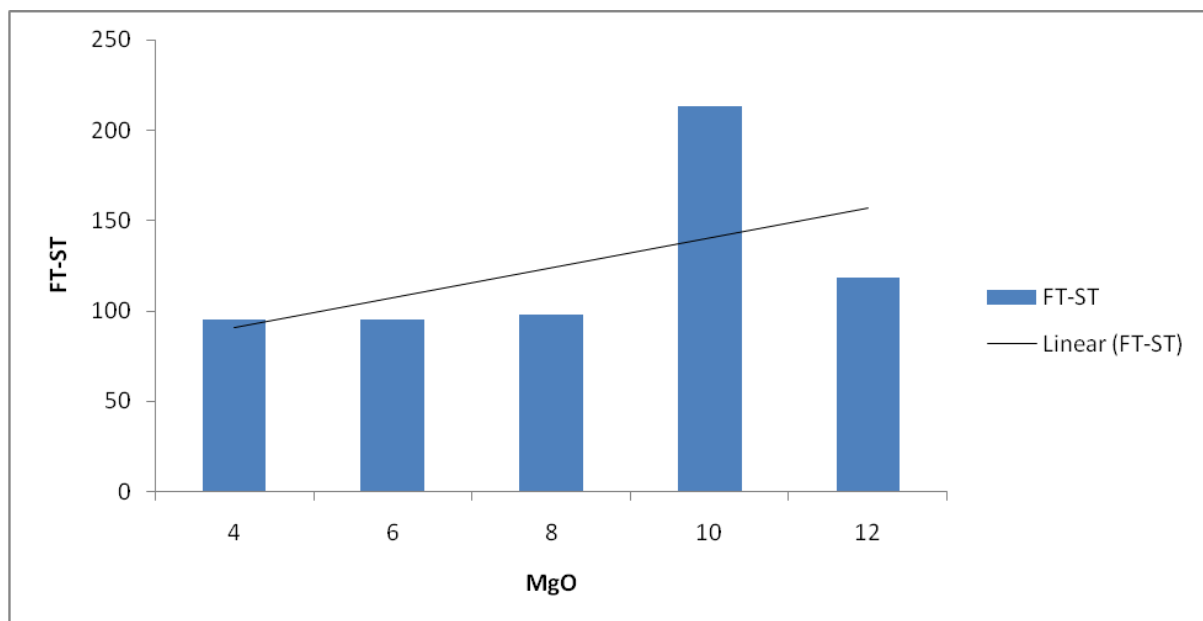
**Figure.21.** Variation of ST-FT temperatures with MgO content for slag nos. 6-10

Slag no. 7 in the 2nd group of slags seems to be a short slag. But its comparison is identical to slag no. 3, which is not a short slag. This variation of results between two identical slags may be a result of experimental error. A report of the experiment reveals that an accurate measurement of characteristic temperature in the case of slag no. 7 could not be had due to excessive bubble formation. It is reported that bubble formation conformed till 1370°C and a correct deformation of FT could not be done. Thus in all probability this step also has a difference between FT and ST in excess of 100°C. However, it is also revealed slag no. 10 in

this category, all steps are very close to being short slags. It can thus be concluded that variation at MgO from 7.4% to 10.2% at C/S ratio of 1.01 may be reported to the Blast Furnace process of iron making.



**Figure.22.** Variation of characteristic temperatures with MgO content for slag nos. 11-15



**Figure.23.** Variation of ST-FT temperatures with MgO content for slag nos. 11-15

Most of the slags in the next category with variation of MgO corresponding to theoretical values for save of the academic interest are short slags. The experimental data reveals that at C/S ratio of 1.1 variation of MgO between 4 to 8 percent may be considered beneficial.

On the light of the above, it can be concluded that through the synthetic slags show same

trand of variation, especially for the slags with c/s ratio of 1.01 with variation of MgO from 7.4% to 10.2% and for slags with c/s ratio 1.1 and MgO variations from 4 to 8 percent, these slags don't exhibit any definite trend with variation of composition. However, in the phase 3 set of experiments pellets may be provided with c/s ratio and MgO variations as noted above.

### **3. Phase III experiments**

#### **Iron Ore Pellets with different extent of reduction**

The chemical analysis of iron ore and other raw materials used for producing the pellets is given in Table no.9. The chemical analysis of the pellets cured at 950°C for 2 hours is given in table no. 10. The tumbler index of the pellets is found to be 94 and the abrasion index is found to be 0.6.

*Table: 9. Chemical analysis of Iron ore and other raw materials*

	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Fe	FeO	CaO	MgO	LOI	SiO <sub>2</sub>	Na <sub>2</sub> O	K <sub>2</sub> O
Iron ore fines	3.75%	0.5%	86.5%	60.45%	77.7%	---	---	6%	2.5%	---	---
Lime stone	0.01	0.22	1.28	---	----	50.35	3.3	43	1.6	---	----
Bentonite	7.6	0.2	13.66	----	----	----	3.96	18	56.44	0.3	0.03
Mgnesite	----	---	0.1	----	---	0.9	97	1.95	----	----	----
Dunite	1.64	0.22	7.7	---	----	0.4	49.5	3	37.39	0.07	0.11
Dolomite	1	---	0.1	----	----	32.05	20.1	45.7	0.95	----	----

*Table no. 10. Chemical composition of pellets after curing at 950°C for two Hrs*

Chemical composition	wt %
Fe <sub>2</sub> O <sub>3</sub>	63.66
SiO <sub>2</sub>	5.46
CaO	5.54
MgO	11.4
TiO <sub>2</sub>	0.44
Al <sub>2</sub> O <sub>3</sub>	13.5

As discussed in the “experimental” section the pellets with reduction % of 10, 50 and 65 are considered for flow characteristics measurements.

The characteristics temperature of the pellets is provided in table no. 11.

Table-11. %Reduction Vs characteristics temperature

% Reduction	Characteristic temperature			
	IDT	ST	HT	FT
10%	1180	1360	1524	1566
50%	1195	1424	1470	1583
65%	1284	1452	1494	1540

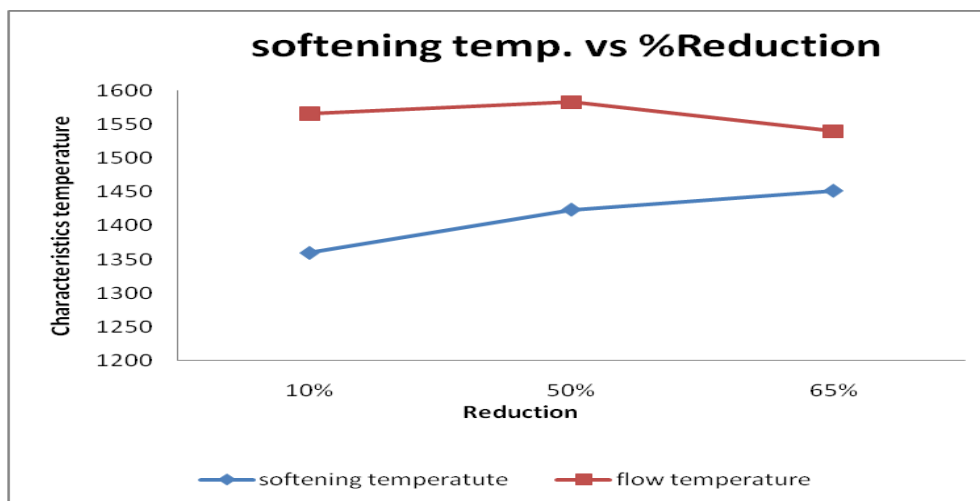


Figure.24. - %Reduction Vs characteristics temperature

Fig no.24. presents the variation of ST and FT and the variation at FT and ST as a function of the extent of reduction. Fig no. 25 the SEM micrographs of polished surfaces of the reduced pellets and also the EDX detail to show the phase /compositional variations are presented separately for the respective samples. The XRD plots for the reduced samples are presented in figure no. 26.

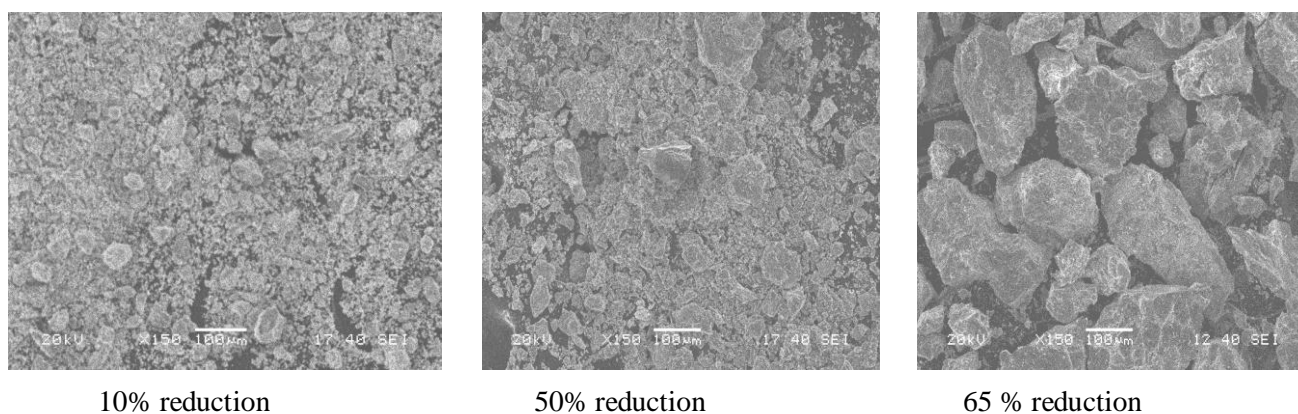
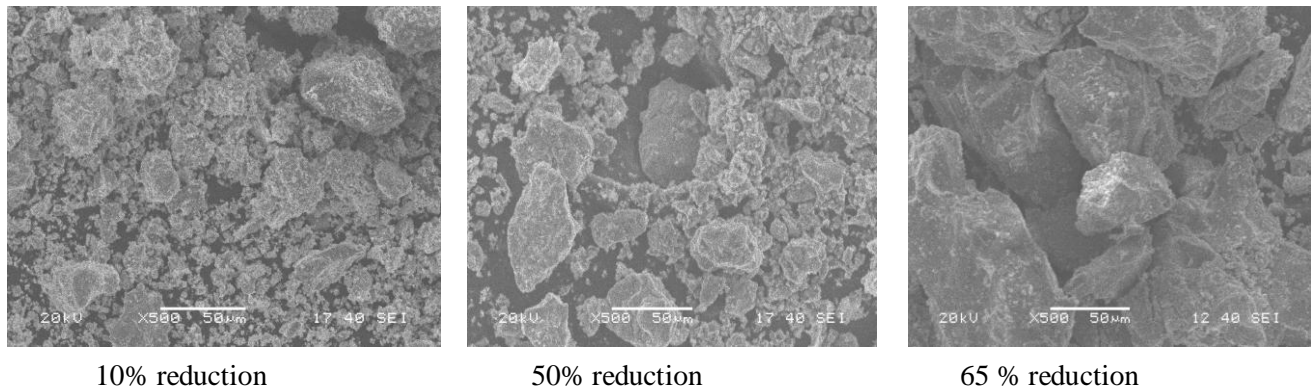


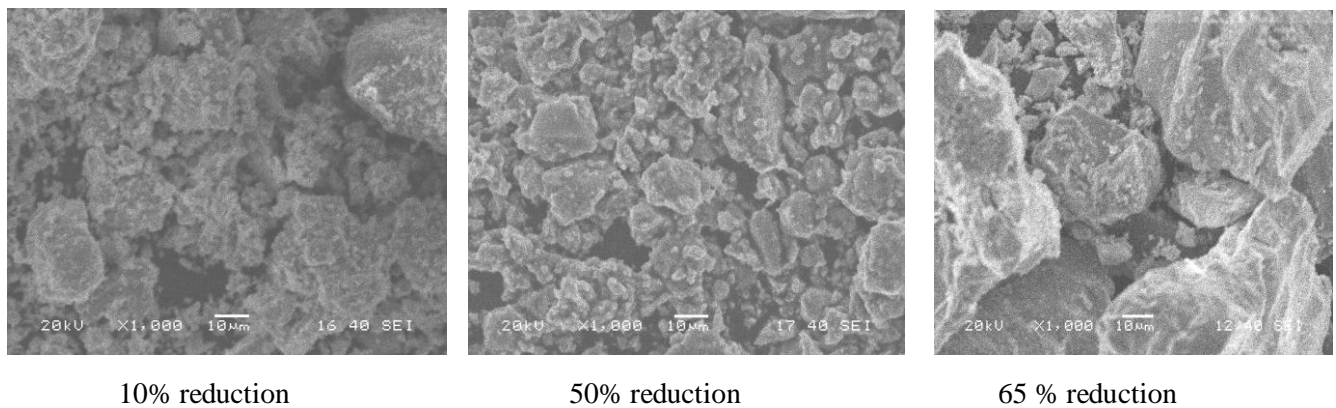
Fig no.25 (a) SEM micrographs at 150 magnifications



10% reduction

50% reduction

65 % reduction

**Fig no.25 (b) SEM micrographs at 500 magnifications**

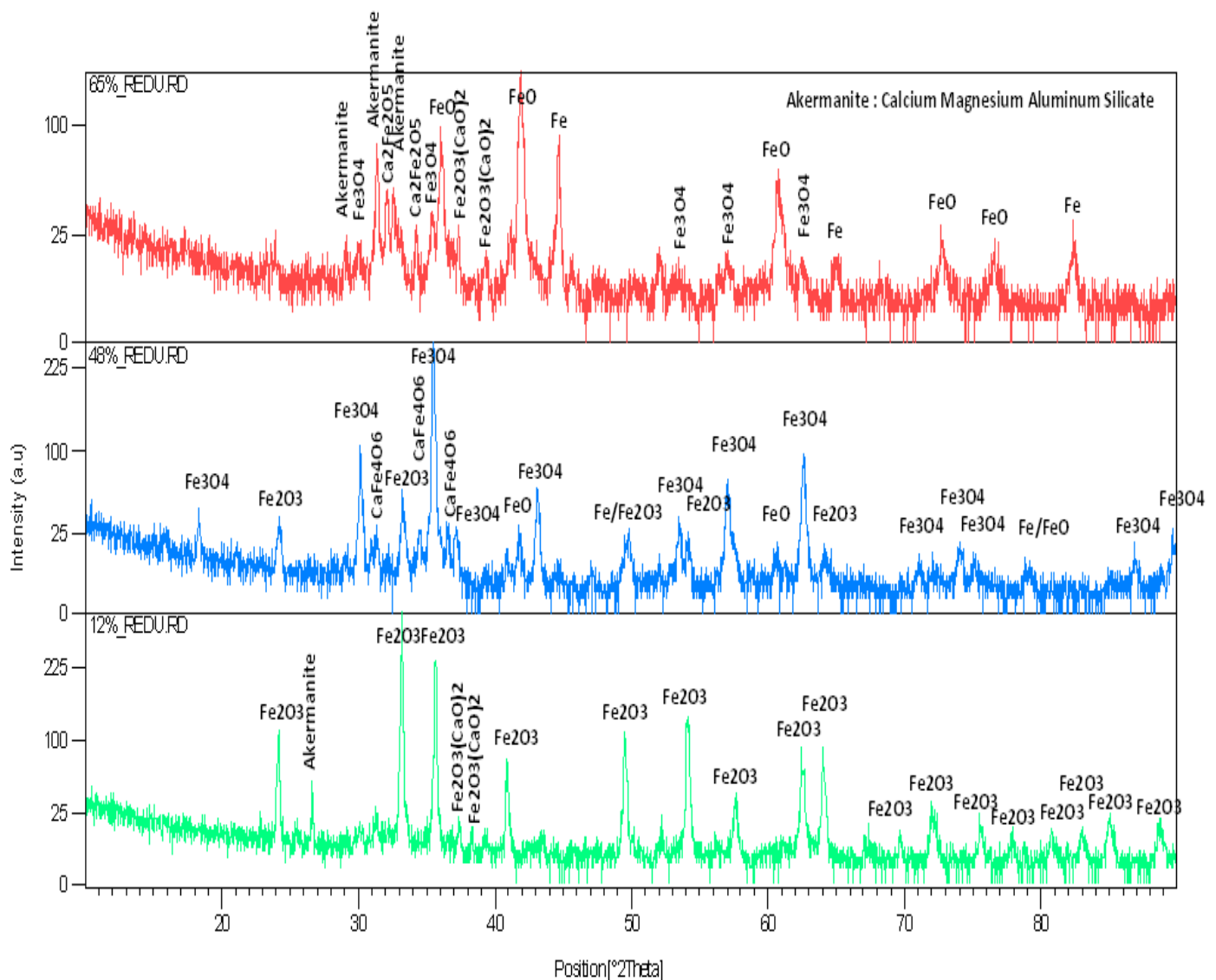
10% reduction

50% reduction

65 % reduction

**Fig no.25 (c) SEM micrographs at 1000 magnifications**

The characteristics temperature shows a definite trend of variations with the variations of the degree of reduction. It can be clearly seen that as per degree of reduction increases the ST increases. This means an increased degree of pre reduction or iron ore softening temperature increases, resulting in the lowering of the cohesive zone which is advantageous. However, the definite conclusion will need further experimentation with various degree of pre reduction of the iron bearing materials. It is also observed that when the degree of reduction is in excess of 50 %, The FT actually decreases promoting the formation of a narrow cohesive zone which is advantageous. However, a definite conclusion will need further experimentation with various degree of pre reduction of the iron bearing materials it is also observed that when the degree of reduction is in excess of 50 %, the FT actually decreases promoting the formation of a narrow cohesive zone. The lowering of the FT may be due to the formation of a definite result as a result of treating the pellets at an elevated temperature for prolonged lengths of time.



**Fig no.26** The XRD plots for the 10% reduction, 50% reduction and 65% reduction samples

The XRD plots, the Edax data and the SEM micrographs the above. The SEM micrographs established the specimen are subjected to different degree of reduction the 50% reduced samples shows a sort of agglomeration and pores formation while this effect is more clear in the SEM pertaining to 65% reduction. Here the grains are no more dispersed fine grains as in 10% reduction but are somewhat coarsened. The micrograph also shows FeO (White portions revealing the higher degree of reduction associated with it).

The XRD plots reveal the formation of complex oxides with higher degree of reduction promoting the formation of a multi –oxide – phase, namely the slag. Whereas the XRD plot pertaining to 10% reduction shows  $\text{Fe}_2\text{O}_3$  peaks the 50% reduced sample reveals  $\text{Fe}_3\text{O}_4$  peaks

and few FeO peaks and the XRD plot for 65% reduced sample exhibits some ‘Fe’ peaks along with ‘FeO’ peaks, Fe<sub>2</sub>O<sub>3</sub> peaks are almost absent. Thus it is established that a higher degree of reduction enhances the formation of the slag and closely monitored extents of reduction will generate very favorable situations in the Blast furnace however such a definite conclusion need further experimentation.

# CHAPTER- 4

---

## CONCLUSION



## CHAPTER-4 CONCLUSIONS

### Conclusions

1. The flow characteristics of blast furnace slags depends on the chemical composition of the slag high c/s ratio coupled with high MgO content (C/S ratio 1.049 to 1.078 and MgO 10.09 to 11.01%) resulting in a short slag.
2. Synthetic slags do not show a definite trend of variation with the variation of composition may be due to the absence of minor constituents in the slag which are not considered while preparing the synthetic slags this may also be due to the temperature and pressure conditions prevailing in the blast furnace however slags with 1.1 C/S ratio and MgO content varying from the 4-8% may be of interest.
3. Higher degrees of pre reduction established a favourable situation in the blast furnace, lowering the FT and increasing the ST for the range of compositions studied. However, a definite conclusion needs more experiments.

### Future work

Tremendous scope is there for further experimentation. Pellets with various degree of pre reduction must be examined. Also composition of the pellets may be varied for experimentation in line with the data as obtained from phase one and phase two set of experiments .

**References:**

- 1) Book: Gupta S. S., and Chatterjee A., “Blast Furnace Iron Making”, SBA Publication.
- 2) Principle of Iron making By A.K. Biswas.
- 3) Book: Tupkary R. H., and Tupkary V. R., “An introduction to modern Iron Making”, Khanna Publication.
- 4) LEE, Y.S., KIM, J.R., YI, S.H. and MIN, D.J; “Viscous behaviour of CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO-FeO slag”: *VII International Conference on Molten Slags Fluxes and Salts*, The South African Institute of Mining and Metallurgy, 2004.
- 5) Y.S.Lee, J.R.Kim, S.H.Yi and D.J.Min; “viscous behavior of CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO-FeO slag”. *Proceedings of VIII international conference on molten slag, fluxes and salts*, The South African Institute of Mining and Metallurgy, 2004, p.225.
- 6) JIA, J.-Y., BAI, C.-G., QIU, G.-B., CHEN, D.-F., and XU, Y; “Calculation models on the viscosity of Cao-SiO<sub>2</sub>-TiO<sub>2</sub> slag system”, *VII International Conference on Molten Slags Fluxes and Salts*, The South African Institute of Mining and Metallurgy, 2004. 52
- 7) Verma, R.K.; Ray, H.S; Gosh, A.; Singh, R.N. and Dharnipalan, S.; *Transactions of Indian Institute of Metals*, Vol. 32. August 1979.
- 8) BQSF; “Report of the Visit of the Slag Study Group of the Japan Iron and Steel Federation of BQSF”, British Quarrying and Slag Federation Limited, June 29, 1978.
- 9) <http://www.steel.org/AM/Template.cfm?Section=Articles3&TEMPLATE=/CM/ContentDisplay.cfm&CONTENTID=25317>, retrieved on 04:05:10
- 10) Paulo Nogueira, Richard Fruehen; “Blast Furnace Burden Softening and Melting Phenomena”- Part I Pellet Bulk Interaction Observation, *Metallurgical and Materials Transactions B*. Vol 35B, 2004, pp.829. 54
- 11) P.F.Noguira and R.J Fruehen; “Blast Furnace Softening and Melting Phenomenon” – Melting Onset in Acid and Basic Pellet: *IUSS-AIME Iron making Conference*, 2002, pp.585.
- 12) U. K. Mohanty: Ph. Dr. Dissertation, R. E. College, Rourkela, (2000).
- 13) U.K. Mohanty, R.C. Behera: *ISIJ Int.*, Vol. 43 (2003), No. 12.
- 14) *IUPAC Compendium of Chemical Terminology*, 2nd Edition, 1997.
- 15) Noritaka Saito, Naoto Hori, Kunihiko Nakashima and Katsumi Mori, *Metallurgical and Materials Transactions*, Volume 34B, October 2003.

- 16) Nakamoto Masashi, Tanaka Toshihiro, Lee Joonho and Usui Tateo, ISIJ International, Vol. 44, No. 12, 2004, pp. 2115-2119.
- 17) Seok Seong-Ho, Jung Sung-Mo, Lee Young-Seok and Min Dong-Joon, ISIJ International, Vol. 47, No. 8, 2007, pp. 1090-1096.
- 18) German Industrial Standard 51730.
- 19) Taskanen P.A., Huttunen S.M., Mannila P.H. and Harkki J.J., *Ironmaking & Steelmaking*, 29, 2002, pp. 281-286.
- 20) Muller M. Erwee, Southern African Pyrometallurgy, 2011.
- 21) Osborn E. F. et al., Process Blast Furnace, Coke Oven and Raw Materials Conference, 1953.
- 22) Snow R. B., Proc., AIME, 1962.
- 23) Ohno A. and Ross H. U., Can. Metall. Q., 2, 1963, p 243.
- 24) Muller J. And Erwee M., Southern African Pyrometallurgy, 2011.
- 25) Clixby G., Softening & Melting of super fluxed sinters and acid pellets, BSC, Ironmaking Session, Tesside Laboratories, International Report, 1979.
- 26) Shankar Amitabh, Gernerup Marten, Lahiri A.K, Seetharaman S., Experimental Investigation of the Viscosities in CaO-SiO<sub>2</sub>-MgO-Al<sub>2</sub>O<sub>3</sub> and CaO-SiO<sub>2</sub>-MgO-Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> Slags, Metallurgical and Materials Transactions B, 38 (6), pp. 911-915.
- 27) B.Ozturk and R.J.Fruehan: "Kinetics of the Reaction of SiO (g) with carbon saturated Iron": Metall.Trans.B. Vol.16B, 1985, p.121.
- 28) B.Ozturk and R.J.Fruehan: "The Reaction of SiO (g) with liquid slags": Metall.Trans.B. Vol.17B, 1986, p.397.
- 29) S.Z. Li, Jian Cheng, W.Q. Ma: „a method to forecast the cohesive zone of the blast furnace". Materials Science Forum, 2004, 471-472, 358.